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PREFACE TO THE SECOND EDITION.

In this edition some changes and additions have been made, but the original plan of the book has been retained. In an appendix a few suggestions are given about the form of record to be kept by the student. Some hints are also given to aid the student in writing the more difficult of the chemical equations. The endeavor has been made to keep the help given, strictly within the limits of what the student has a right to expect at the stage in his progress when qualitative analysis is usually begun.

Hypochlorous and nitrous acids have been added at the suggestion of Professor T. H. Norton, and heating with concentrated sulphuric acid, in the preliminary examination, at the suggestion of Professor E. J. Bartlett.

June, 1890. **W. A. N.**

PREFACE TO FIRST EDITION.

Two reasons have led to the writing of this book. One has been the desire to give to my classes a scheme for qualitative analysis in which points where a beginner is liable to make mistakes are especially guarded by careful and explicit directions for procedure. In this respect the book is the result of considerable experience with classes in the laboratory, where especial pains have been taken to discover the reasons for errors on the part of ordinary students. Qualitative analysis is of no value unless it leads to *certain* results, and the beginner needs to have it impressed upon his mind that certainty can only be attained by performing each operation in exactly the right way. In the author's opinion it is better to teach the student the right way at first, rather than to give only an outline and expect him to fill in the details of manipulation for himself. Every one who has had experience in the matter knows, however, that no amount of careful direction in a text-book can supply the place of constant watchfulness on the part of the instructor.

The second reason for the book has been the desire to connect the reactions given by way of a study of the elements before actual analysis more closely with the course of analysis itself. With this end in view, the reactions given for the student to try before taking up the actual analysis are chosen and arranged in each case with reference to their immediate use in the separation and detection of the elements under consideration. As a result, the preliminary reactions given are fewer in number than is usually the case. These reac-

PREFACE TO FIRST EDITION.

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The second reason for the book [illegible] to connect the reactions given [illegible] before actual analysis more [illegible] sis itself. With this end in view [illegible] student to try before taking [illegible] and arranged in [illegible] tions are supplemented, however, by the tables given at the close of Part I. For the idea embodied in these tables, and, indeed, for most of the matter which they contain I must acknowledge my indebtedness to Biedermann's "Chemiker-Kalender" for 1887.

The plan to be followed in using this book is largely implied in the text. In the first part of the work it is my custom to have the student perform the preliminary experiments with the metals of a group, writing the equations representing the reactions involved and keeping a careful record of his work. A mixture containing all of the metals of the group is then given and he is required to analyze it, keeping in some systematic form a record of each reagent used and of the results obtained. When the analysis is complete he is required to explain his record and give the reason for each operation. Then mixtures containing part of the metals of the group are given. After completing the study of the metals in this way, simple salts containing one metal and one acid are given for analysis and then general mixtures of various kinds.

The author will be very glad of any corrections or suggestions for improvement which may occur to any one who uses the book.

TERRE HAUTE, IND., August, 1887.

W. A. N.

INTRODUCTION.

The **qualitative analysis** of a substance consists in separating each of the elements which it contains in the free state or in the form of some compound which is characteristic and easily identified. Separation in the free state is comparatively rare either in qualitative or quantitative analysis.

In the course of a qualitative analysis it is usually necessary to transform an element into a number of different compounds, successively, either because the compound first formed is so similar to some compound of another element that it cannot be identified with certainty, or because it is necessary to separate the element from others with which it is mixed or combined.

A qualitative analysis to be successful must prove with *certainty* that the elements sought are present or that they are absent. The latter is as important, and in many cases it is more important than the former. Success can only be attained by the exercise of care in all operations and by the most scrupulous cleanliness.

One who attempts to analyze a substance by following mechanically a scheme which is laid down may sometimes succeed in finding the substances which he seeks, but he is sure to fail in many cases. In order to avoid constant mistakes the chemist must understand thoroughly the object of every operation and the effect of the operation upon every substance present in the mixture with which he is working.

For this reason it is best to perform the operations of analytical chemistry, at first, with substances of known composition, studying carefully the effect upon these of operations which are afterwards to be used for the detection of unknown substances. It should also be an invariable rule, when the chemist is in doubt about a test which he is applying, to apply the same test to some substance which he knows to contain the substance sought and to compare the effect with that upon the substance under examination.

Precipitation is more often used than any other means for the separation and detection of substances. A **precipitate** is a substance insoluble in the liquid which is present. If the solutions of two salts containing two metals and two acids are mixed, the resulting solution probably contains, at first, the four possible salts which may be formed from the substances present. Thus, if solutions of barium chloride and copper sulphate are mixed, there are probably present at first in the resulting solution four salts,—barium chloride, copper sulphate, copper chloride and barium sulphate. If, however, one of the four (in this case barium sulphate) is insoluble in the liquid present, it will separate and its formation will continue until either all of the barium or all of the sulphuric acid is removed from the solution.

Filtration consists in pouring a liquid in which a precipitate is suspended, upon a filter and allowing the liquid to run through. The latter is then called the **filtrate.**

Decantation consists in pouring off a liquid from which a precipitate has been allowed to settle. It is frequently advisable to combine decantation and filtration.

A precipitate is **washed** for the purpose of removing the liquid which is mechanically held within its mass. Washing may be performed by decantation or by repeatedly pouring water upon the precipitate as it lies on the filter; unless the precipitate is heavy and settles rapidly, washing upon the filter is usually most effective. In almost all cases a precipitate can be washed more rapidly with hot than with cold water. For special reasons, however, some precipitates must be washed with cold water. The success of an analysis frequently depends upon the thorough washing of precipitates, but in most cases the washing need not be so thorough as is necessary in quantitative analysis. In most cases a precipitate will be washed sufficiently for qualitative purposes by filling the filter four or five times with hot water and allowing the liquid to run through completely after each addition. It is frequently advisable to remove the first filtrate before beginning to wash a precipitate, as the washings sometimes pass through turbid even when the first filtrate is clear.

Reagent is the general name applied to any substance used in the course of an analysis. The most common reactions made use of in analysis are of the nature of double decompositions and may be expressed by the following form: $\frac{\text{A}}{\text{C}}\Big|\frac{\text{B}}{\text{D}}$, or illustrated by the action of hydrochloric acid on silver nitrate, thus: $\frac{\text{Cl}}{\text{Ag}}\Big|\frac{\text{H}}{\text{NO}_3}$. The horizontal line divides the two substances present before the solutions are mixed; the perpendicular line divides the two substances formed by the reaction of the first two upon each other.

The Blowpipe is used in oxidizing or reducing substances on charcoal, in the preparation of borax and other beads and in tests of fusibility and flame coloration. The most convenient flame is obtained by introducing into the Bunsen burner a brass tube which is narrowed to a slanting, slit-like opening at the top and which reaches to the bottom of the burner, excluding the air. The **oxidizing flame** (O. F.) is produced by placing the blowpipe within the flame and blowing rather strongly. Substances placed at the extreme tip of the flame will be strongly heated by it and can at the same time obtain oxygen from the air. The **reducing flame** (R. F.) is obtained by placing the blowpipe at the edge of the flame and blowing less strongly.

Substances placed in the luminous tip of the flame meet with an excess of carbon and are reduced by it.

Many of the blowpipe reactions can be performed equally well with the Bunsen flame alone. The outer edge one-third of the way up is an oxidizing flame, while the luminous tip just above the central dark zone is a reducing flame.

PART I.

DETECTION OF METALS.

GROUPS OF THE METALS.

For the purposes of qualitative analysis the metals are divided into five groups in accordance with their deportment toward various reagents. A brief statement of the leading characteristic of each group is given here while fuller statements are given in the discussion of each group.

Group I.—Metals whose chlorides are insoluble in acids: Lead (slightly soluble), Silver, Mercury (ous).

Group II.—Metals whose sulphides are insoluble in cold, dilute acids. Mercury (ic), Lead, Bismuth, Copper, Cadmium; Arsenic, Tin, Antimony, Gold, Platinum.

Group III.—Metals whose hydroxides or sulphides are insoluble in ammonium hydroxide. Chromium, Aluminum, Iron; Cobalt, Nickel, Manganese, Zinc.

Group IV.—Metals whose carbonates are insoluble in ammonium chloride. Barium, Strontium, Calcium.

Group V.—Metals whose chlorides, sulphides and carbonates are soluble in ammonium chloride. Magnesium, Sodium, Potassium, Ammonium.

GROUP I.

Hydrochloric Acid Group.

To this group belong **Lead, Silver, and Mercury in mercurous salts.**

The chlorides of the metals of this group are insoluble in acids, with the exception of lead chloride, which is very sparingly soluble. Enough lead always remains in solution so that, if present in small amount, it may not be found at all in the first group.

The sulphides of the metals of the group are insoluble in cold, dilute acids and in ammonium sulphide.

PRELIMINARY EXPERIMENTS.

In performing the experiments with each metal the student should keep a concise record of his work, and afterwards in analyzing a solution containing the metals of the group, he should refer to his record and *be sure that he understands thoroughly the effect of every reagent which he uses, and the object of every operation which he performs.*

Lead.—Take 2 cc. of a solution of lead nitrate or lead acetate in a test tube, add a little dil. HCl, shake vigorously and allow the white precipitate of $PbCl_2$ to settle. Decant the liquid. Boil the precipitate with water, pour off the solution, boil again with water and repeat until the lead chloride is completely dissolved. Test different portions of the solution with H_2S, dil. H_2SO_4 and $K_2Cr_2O_7$. The precipitates are PbS, $PbSO_4$ and $PbCrO_4$.

Silver.—Take 2 cc. of a solution of silver nitrate. Add dil. HCl, shake, allow the white, curdy AgCl to settle, decant, boil the precipitate with water, decant through a filter and test the filtrate for Ag with H_2S. Add to the AgCl remaining in the test tube, NH_4OH and shake. To the solution, which contains the silver as ammonio-silver chloride, $2\ AgCl. 3\ NH_3$, add dil. HNO_3. The precipitate is AgCl.

Explain its formation. Filter, and expose the precipitate to diffused sunlight.

Mercury in mercurous salts.—Take 2 cc. of a solution of mercurous nitrate, add dil. HCl, shake, decant, boil the HgCl three times with about 10 cc. of water, decanting each time, to remove mercuric salts, then boil a fourth time, decant through a filter and test the filtrate for Hg with H_2S. Add NH_4OH to the HgCl remaining in the test tube. The mercurous chloride is changed to black mercurous amido chloride, Hg_2NH_2Cl. Pour off the ammonia and wash the precipitate once by decantation. Dissolve by boiling with dil. aqua regia. Add $SnCl_2$. The mercury dissolves in the aqua regia as mercuric chloride and is reprecipitated by the stannous chloride as mercurous chloride, or, if the $SnCl_2$ is in excess, as Hg. The latter appears as a gray precipitate.

ANALYSIS.

I.—Hydrochloric Acid Group.

(1). If the original solution has an alkaline reaction, reference must be made to page 60.

Add to the solution one or two drops of dil. HCl.

a. *No precipitate is formed.* Silver and mercurous mercury are absent. Pass on to (5).

b. *A precipitate may contain* $PbCl_2$, AgCl, HgCl, SbOCl and BiOCl. The two last will re-dissolve in an excess of HCl. Add HCl drop by drop until after vigorous shaking an additional drop produces no further precipitate. A large excess of HCl is to be avoided, as it would interfere with the precipitation of Group II. Filter and wash the precipitate on the filter twice with *cold* water. Treat filtrate and washings by (5).

(2). **Lead.**—Heat about 5 cc. of water to boiling and pour over the precipitate. Divide the filtrate obtained in two portions; to one add dil. H_2SO_4. A white pulverulent precipitate is $PbSO_4$. To the other portion add $K_2Cr_2O_7$. A yellow precipitate is $PbCrO_4$.

(3). **Silver.**—If lead has been found, pour hot water over the precipitate repeatedly until it is completely removed. To the precipitate on the filter add a few drops of NH_4OH, taking care to drop it in such a way that the whole precipitate is moistened with it, but do not use more than 5 or 10 drops of the reagent unless the precipitate is very large. To [illegible] filtrate add dil. HNO_3 to acid reaction.* The filtrate is sometimes turbid from the presence of a basic lead salt, $PbOHCl$, but this is soluble in HNO_3 while the silver is precipitated, as $AgCl$. If much mercury is present and silver is not found here, it is best to test for silver, any residue which remains on dissolving the mercurous-amido-chloride in aqua regia.

(4). **Mercurous Mercury.**—If the precipitate blackens on treatment with NH_4OH, the formation of mercurous-amido-chloride, Hg_2NH_2Cl, is indicated. Make a hole in the bottom of the filter and rinse the precipitate into a test tube. Allow to settle. Decant the liquid completely. Dissolve the precipitate by boiling with a very little dilute aqua regia. Dilute the solution with an equal volume of water, filter, if any $AgCl$ has remained undissolved, and test the solution or filtrate for mercury by adding $SnCl_2$.

GROUP II.

Hydrogen Sulphide Group.

The sulphides of the metals of this group are insoluble in dilute acids. The group is divided into two sub-groups:

Sub-Group A.—To this belong **Mercury in mercuric salts, Lead, Bismuth, Copper** and **Cadmium.**

2 3 4 5

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*In every case like this, test the solution with litmus paper, after adding the reagents and *thoroughly shaking the liquid.*

The sulphides of the metals of this sub-group are insoluble in yellow ammonium sulphide.

Sub-Group B.—To this belong **Arsenic, Antimony, Tin, Gold** and **Platinum.** The sulphides of the metals of this sub-group are soluble in yellow ammonium sulphide and are reprecipitated *as sulphides* on adding dil. HCl to the solution.

PRELIMINARY EXPERIMENTS.

Mercury in Mercuric Salts.—Take 2 cc. of mercuric chloride, dilute to 10 cc., pass H_2S slowly as long as a change is observed in the color of the precipitate, warm, allow to settle and wash three or four times by decantation. Try to dissolve the HgS, first by boiling with dil. HNO_3, and then by boiling with aqua regia. The mercury dissolves as $HgCl_2$. Cool and add $SnCl_2$. The precipitate is HgCl mixed with Hg.

Lead.—Take 10 cc. of a solution of $PbCl_2$, warm, pass H_2S, allow the PbS to settle, decant, wash, boil the precipitate with 1 cc. of dil. HNO_3. To the solution containing lead nitrate, add a few drops of dil. H_2SO_4. If this does not give enough of a precipitate of lead sulphate to work with, evaporate nearly to dryness in a porcelain dish, rinse back into a test tube, allow to settle, wash, dissolve in a very little alkaline ammonium tartrate and add $K_2Cr_2O_7$. The precipitate is $PbCrO_4$, "chrome yellow."

Bismuth.—Take 2 cc. of a solution of $BiCl_3$. Dilute to 10 cc. This may cause a precipitate of BiOCl. Whether it does or not, warm, pass H_2S, allow the Bi_2S_3 to settle, decant, wash, boil the precipitate with 1 cc. of dil. HNO_3. To the solution of $Bi(NO_3)_3$ add two or three drops of dil. H_2SO_4, then NH_4OH. The precipitate is BiOOH. Filter, wash once. Drop three or four drops of dil. HCl upon the precipitate and allow the solution of $BiCl_3$ to run through into a clean test tube. Add to the solution 10 to 20 cc. of water. The precipitate is BiOCl, bismuth oxychloride.

Copper.—Take 2 cc. of a solution of $CuSO_4$, dilute to 10 cc., warm, pass H_2S, allow the CuS to settle, decant, wash, dissolve in 1 cc. dil. HNO_3 by boiling, add two or three drops of dil. H_2SO_4 to the solution of $Cu(NO_3)_2$ and then NH_4OH to alkaline reaction. The blue color is caused by a cupro-ammonium salt $CuSO_4.4\ NH_3$ or $Cu(NO_3)_2.\ 4\ NH_3$. Add dil. HCl till the deep blue just disappears, then some iron filings and boil for a short time. Filter and test the filtrate with H_2S.

Cadmium.—Take 2 cc. of a solution of $CdSO_4$ or of $Cd(NO_3)_2$, dilute to 10 c.c., warm, pass H_2S, filter, wash. Make a hole in the bottom of the filter and rinse the CdS into a test tube with the least amount of dil. HNO_3 possible. Boil, add to the solution of $Cd(NO_3)_2$ two or three drops of dil. H_2SO_4 and then NH_4OH to alkaline reaction. The $Cd(OH)_2$ which would have been formed on adding NH_4OH to a normal cadmium salt, remains, in this case, dissolved in the excess of NH_4OH. Add HCl to acid reaction, then some iron filings and boil for a short time. Filter and test the filtrate with H_2S. The precipitate is yellow Cd S.

ANALYSIS.

II.—Hydrogen Sulphide Group.

(5). **Precipitation.**—If the solution to be tested for Group II. contains much free acid, it may be necessary to evaporate it nearly to dryness before all of the metals of the group can be precipitated, but usually dilution with water will be sufficient. Warm a *small portion** of the solution to about 70° and pass H_2S through it. If arsenic is suspected to be present, pass the gas slowly for some time and warm the solution repeatedly, passing H_2S each time.

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*The use of a small portion instead of the whole solution, is to avoid the introduction of H_2S into the latter in case Group II. should prove to be absent. If metals of the group are known to be present, of course the whole solution may be treated at once as directed in c.

a. *No precipitate forms even after diluting with four volumes of water and testing again with* H_2S. Group II. is absent. Treat the principal solution by (**16**).

b. *A fine white precipitate of sulphur is formed.* This is caused by ferric iron or some other oxidizing agent. If accompanied by a change of color from reddish yellow to green, the presence of a chromate is indicated. Group II. is absent. Treat the principal solution by (**16**).

c. *A colored precipitate is formed.* This may contain HgS, PbS, Bi_2S_3, CuS, CdS ;| As_2S_3, Sb_2S_3, SnS, SnS_2, Au_2S_3 and PtS_2. Warm the whole solution, pass H_2S long enough for complete precipitation, filter and wash the precipitate three or four times with hot water. Dilute a small portion of the filtrate with four volumes of water and pass H_2S to make sure that the metals of the group are entirely removed. Afterwards examine the solution from which Group II. has been entirely precipitated, by (**16**). If nothing is known about the nature of the precipitated sulphides, test a small portion as directed in (**6**). If it is already known that only Sub-Group A is present, treat the sulphides at once by (**7**). If only Sub-Group B is present, treat them as directed in (**13**). If metals of both Sub-Groups are present, treat the sulphides at once as directed in (**6**), bb.

(**6**). **Separation of Sub-Group A and Sub-Group B.**—By means of a glass spatula* put a small portion of the precipitate into a test tube, add 5 or 10 drops of yellow $(NH_4)_2S$ and a little water and warm gently.

a. *The precipitate dissolves completely.* Sub-Group A is absent. Treat the main precipitate by (**13**).

b. *A residue is left.* Sub-Group A is present. Filter, and add dil. HCl to the filtrate to acid reaction.

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* Easily made by softening half an inch of glass tubing in the flame and pinching it with forceps. It should then be held in the flame again and annealed.

aa. *A pure white precipitate of sulphur is obtained.* Sub-Group B is absent. Treat the main precipitate by (**7**).

bb. *A colored precipitate is obtained.* Sub-Group B is present. Put the whole precipitate into a test tube by means of a glass spatula, add 5 to 10 cc. of yellow $(NH_4)_2S$, warm gently for some time and allow to settle. If the precipitate settles leaving a clear yellow liquid above,* pour the whole upon a filter and allow the liquid to run through as completely as possible. Treat the filtrate by (**12**). Wash the precipitate twice with hot water, to which one-tenth volume of $(NH_4)_2S$ has been added, using about 10 cc. each time and allowing the liquid to run through completely after each addition. If the washings are of a pure yellow color, add them to the first filtrate. If the precipitate containing Sub-Group A is sufficient in quantity, transfer it to a test tube by means of a glass spatula and treat it by (**7**). Otherwise make a hole in the filter and rinse it into a test tube with water, just acidify with dil. HCl, warm, pass H_2S, filter, wash three or four times with hot water and treat the precipitate by (**7**). By this treatment the precipitate is usually rendered compact so that it will settle, while the precipitate which has been treated with $(NH_4)_2S$ will not.

(**7**). **Mercuric Mercury.**—Introduce the precipitate, which may contain HgS, PbS, Bi_2S_3, CuS, and CdS, into a test tube with a spatula (or by washing through a hole in the filter, allowing to subside and pouring off the water completely). Add from 1 to 5 cc. of dil. HNO_3, boil as long as anything dissolves, dilute slightly, and pour the liquid off through a filter leaving any residue in the test tube. Treat the filtrate by (**8**). The residue may contain black HgS or white $(HgS)_2\,Hg(NO_3)_2$ or $PbSO_4$, all of which are heavy and settle, or sulphur which is yellow and floats. Enough lead always passes into solution in the HNO_3 for its detection, if present. Boil the residue with 1 or 2 cc. of dil. aqua regia, dilute, filter, if necessary, and to the solution or filtrate add $SnCl_2$. The mercury dissolves as mercuric chloride and is precipitated as mercurous chloride (calomel), or as metallic mercury.

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*If the liquid remains turbid at the top or is colorless, enough $(NH_4)_2S$ has not been used or the reagent has decomposed by standing.

(**8**). **Lead.**—To the filtrate containing Pb, Bi, Cu, and Cd, as nitrates, add ½ cc. of dil. H_2SO_4 and evaporate in a porcelain dish till only a few drops remain, but not after fumes of H_2SO_4 appear. Rinse the precipitate of $PbSO_4$ into a test tube with water, adding dil. H_2SO_4, if necessary, to keep Bi in solution, allow to settle and filter quickly, leaving the precipitate in the test tube. Treat the filtrate by (**9**). Wash the precipitate by decantation, dissolve it by warming with a few drops of alkaline ammonium tartrate and add $K_2Cr_2O_7$ in sufficient amount. A yellow precipitate is $PbCrO_4$, "chrome yellow."*

(**9**). **Bismuth.**—To the solution, containing Bi, Cu and Cd as sulphates, add NH_4OH to alkaline reaction. A precipitate is BiOOH. Filter and treat the filtrate by (**10**). Wash the precipitate once, drop *two or three drops* of dil. HCl upon it and allow the solution of $BiCl_3$ to run through into a clean test tube. Add 10 to 20 cc. of water. A white precipitate is BiOCl, bismuth oxychloride.

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*Another method is to warm the $PbSO_4$ with a few drops of $K_2Cr_2O_7$, which will convert it into $PbCrO_4$. The precipitate is then washed by decantation and a few drops of NaOH are added. The $PbCrO_4$ will dissolve to a clear yellow fluid and may be reprecipitated by acetic acid.

(10). **Copper.**—If the filtrate is of a blue color, copper is present as $4NH_3.CuSO_4$. In any case, treat the solution by (11).

(11). **Cadmium.**—If copper is absent, add HCl just to acid reaction, and pass H_2S through the solution. A yellow precipitate is CdS. If copper is present, add dil. HCl till the deep blue color disappears, then some iron filings, boil for a short time, filter and test the filtrate with H_2S. A yellow precipitate is CdS.*

Sub-Group B.

To this Sub-Group belong Arsenic, Antimony, Tin, Gold and Platinum. The sulphides of the metals of the Sub-Group are soluble in yellow ammonium sulphide and are reprecipitated as sulphides on adding dil. HCl to the solution.

PRELIMINARY EXPERIMENTS.

Arsenic in Arsenious Compounds.—Use a solution of As_2O_3 in HCl. Precipitate with H_2S, warming the solution. Filter, put the precipitate into a test tube by means of a spatula, add a little $(NH_4)_2S$ and warm gently. The As_2S_3 dissolves as ammonium sulpharsenite,$(NH_4)_3AsS_3$. (Compare with the formula of ammonium arsenite). Add dil. HCl to acid reaction, filter, wash and boil the As_2S_3 obtained with 1 or 2 cc. of conc. HCl. Then add a fragment of $KClO_3$ and boil again. This time the arsenic will dissolve as arsenic acid, H_3AsO_4. Add NH_4OH to alkaline reaction, filter, if necessary, then add $MgSO_4$ and shake vigorously. The precipitate is $MgNH_4AsO_4$.

Arsenious compounds give no precipitate on adding NH_4OH, NH_4Cl and $MgSO_4$. They are oxidized to arsenic

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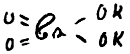
*Another method is to add KCN to the alkaline solution until the blue color is destroyed and then pass H_2S. The cadmium is precipitated as yellow CdS but is often dark colored from the presence of traces of copper or mercury.



acid by free chlorine, or by $K_2Cr_2O_7$ and HCl, or any similar agents giving free chlorine. In this case the chromium is reduced to Cr_2Cl_6 which gives a green solution. Try the reaction and write the equation.

Arsenic in Arsenic Compounds.—Use a solution of arsenic acid, H_3AsO_4. H_2S precipitates from the solution, with some difficulty and on warming, As_2S_3 mixed with S.

NH_4OH, NH_4Cl and $MgSO_4$ give a precipitate of $MgNH_4AsO_4$.

Tin in Stannous Compounds.—Use a solution of $SnCl_2$. Warm, precipitate with H_2S, wash, dissolve the SnS in yellow $(NH_4)_2S$ and add dil. HCl to the solution. The precipitate is now SnS_2. Filter, boil with conc. HCl till H_2S is expelled. The solution now contains $SnCl_4$. Dilute slightly, add an iron nail and warm for a short time. The $SnCl_4$ is reduced to $SnCl_2$. Pour into another tube and add $HgCl_2$. The precipitate is HgCl. See (**4**) and (**7**).

Tin in Stannic Compounds.—Use a solution of $SnCl_4$. Stannic compounds give with H_2S a precipitate of yellow SnS_2. They give no precipitate with HgCl.

Antimony—Use a solution of $SbCl_3$. Warm, precipitate with H_2S, dissolve the Sb_2S_3 in Yellow $(NH_4)_2S$, and add dil. HCl to the solution. Filter, boil the Sb_2S_3 with conc. HCl till H_2S is completely expelled. Divide the solution containing $SbCl_3$ in two portions. Warm one portion with an iron nail. The black precipitate is metallic antimony. Pour the other portion into a porcelain dish, put in it a piece of platinum and lay upon that a piece of zinc, diluting if necessary to bring the solution in contact with the zinc. The black stain on the platinum is metallic antimony. Clean the platinum as directed in (**15**).

Gold.—H_2S precipitates from solutions of gold either Au_2S_3 or Au_2S, which are insoluble in conc. HCl, but are soluble in aqua regia.

A dilute solution of $SnCl_2$ and $SnCl_4$ produces, even in very dilute solutions of gold, a purple red precipitate or coloration which has been called purple of Cassius.

Platinum.—H_2S precipitates from solutions of platinum PtS_2, which is insoluble in conc. HCl, but is soluble in aqua regia.

KCl produces in solutions of platinic salts, if not too dilute, a yellow precipitate of K_2PtCl_6 (or 2 KCl, $PtCl_4$). NH_4Cl produces a similar precipitate.

ANALYSIS.

Group II. Sub-Group B.

(12). Add to the solution of the sulphides of As, Sn, and Sb in yellow $(NH_4)_2S$, dil. HCl to acid reaction, filter and wash. Treat the precipitate by **(13)**. If the original solution contained copper, portions of it will sometimes dissolve in the $(NH_4)_2S$ as a cuprous sulpho-salt and will interfere with the detection of antimony in **(15)**. The copper may be separated by re-dissolving this precipitate in Na_2S which will dissolve only the As, Sn and Sb. Whether copper is present so as to render this necessary, may be determined by testing a few drops of the original acid solution with a clean iron nail, which will cover itself with a coating of metallic copper, if that is present.

(13). Arsenic.—Introduce the precipitate, which may contain As_2S_3, SnS_2 and Sb_2S_3, into a test tube by means of a spatula. Boil with 2 or 3 cc. of conc. HCl, dilute with an equal volume of water, warm, filter hot, leaving a residue of As_2S_3, if present, in the test tube. Treat the filtrate, containing $SnCl_4$ and $SbCl_3$, by **(14)**. Dissolve the As_2S_3 in 1 or 2 cc. (not more) of conc. HCl and a fragment of $KClO_3$. The arsenic dissolves as arsenic acid, H_3AsO_4. Add NH_4OH to alkaline reaction, filter if necessary, add NH_4Cl, then more NH_4OH, if the solution is not strongly alkaline, and $MgSO_4$. Shake vigorously and rub the wall of the test tube with a glass rod to aid in the formation of the crystals of $MgNH_4AsO_4$. The precipitate should be white and crystalline.

(14) **Tin.**—If a yellow or orange precipitate forms in the solution containing $SbCl_3$ and $SnCl_4$ on cooling, boil until H_2S is completely expelled. Divide the solution in two portions. To one portion add a small iron nail and warm gently for several minutes, adding more HCl, if necessary, to cause a vigorous evolution of hydrogen. The nascent hydrogen reduces the antimony to the metallic state and the stannic chloride to stannous chloride. Pour off or filter into another test tube and add $HgCl_2$. A precipitate of HgCl indicates the presence of tin in the solution. Whether the tin was originally present as a stannous or a stannic compound must be determined by adding $HgCl_2$ to a few drops of the original solution. A precipitate insoluble in conc. HCl proves the tin to have been stannous.

(15). **Antimony.**—Pour the other portion of the solution into a porcelain dish, put in it a piece of platinum and lay upon that a piece of zinc, diluting if necessary, to bring the liquid in contact with the zinc. The antimony will be reduced to the metallic state* and will form a black stain on the platinum. A gray stain, which is frequently formed by the reduction of the tin or from impurities in the zinc, must not be mistaken for antimony. The platinum can be cleaned by washing thoroughly and then warming with a little HNO_3 and a drop of ammonium tartrate.

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*If the zinc is left till the evolution of H nearly ceases, the tin will also be deposited, partly on the zinc and partly on the platinum. After carefully pouring off the liquid and removing the zinc, the tin may be dissolved by warming with conc. HCl while the antimony, if much is present, will remain undissolved. The solution, containing $SnCl_2$, may be tested for tin with $HgCl_2$.

GROUP III.

Ammonium Sulphide Group.

To this group belong **Aluminium, Chromium** and **Iron,** which are precipitated as hydroxides by NH_4OH in the presence of NH_4Cl; and **Cobalt, Nickel, Manganese** and **Zinc,** which are not precipated by NH_4OH in presence of NH_4Cl unless phosphoric or some similar acid is present. $(NH_4)_2S$ converts $Fe_2(OH)_6$ into FeS, but has no effect upon $Al_2(OH)_6$ or $Cr_2(OH)_6$.

In presence of phosphates, borates, oxalates, silicates, fluorides or tartrates, **Barium, Strontium, Calcium** and **Magnesium** may be precipitated with this group.

PRELIMINARY EXPERIMENTS.

Chromium.—Use a solution of $K_2Cr_2(SO_4)_4$, "chrome alum." Add NH_4Cl and NH_4OH and warm. The precipitate is $Cr_2(OH)_6$. Add $(NH_4)_2S$. The precipitate is unchanged. Filter, wash with hot water, dissolve in dil. HCl, *nearly* neutralize with Na_2CO_3, add $BaCO_3$, * shake, filter and (test the filtrate for Cr with NH_4OH and warming.) The chromium is precipitated as a basic salt by the $BaCO_3$. Boil the precipitate with NaOH in a porcelain dish, and filter. Save the precipitate. (To the filtrate add dil. HCl to acid reaction, then NH_4OH and boil. If a precipitate is obtained it is due to impurities in the NaOH (silica from the glass, etc.) Notice it carefully and save it to compare with the aluminium precipitate obtained in the same way) Fuse the precipitate, which has been boiled with NaOH, on platinum foil with 2 parts of Na_2CO_3 and 2 parts of $KClO_3$. The chromium is oxidized to K_2CrO_4. Boil the fused mass with water and filter. The yellow color of the filtrate is due to K_2CrO_4.

All salts in which chromium acts as a base give solutions which are green or violet.

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*Shake the bottle. Why?

Chromium as Chromic Acid.—Use a solution of potassium pyrochromate ("bichromate"). Add dil. HCl and pass H_2S. The chromium is reduced to Cr_2Cl_6. All salts of chromic acid give solutions which are red or yellow.

Aluminium.—Use a solution of ordinary alum, $K_2Al_2(SO_4)_4$. Try the same experiments as with chromium, omitting the fusion with Na_2CO_3 and $KClO_3$. Until the precipitate by $BaCO_3$ is boiled with NaOH, the aluminium compounds formed are exactly analogous to the chromium compounds. Then the aluminium dissolves as sodium aluminate, Na_3AlO_3. On acidifying the filtrate, Al_2Cl_6 is formed and then NH_4OH precipitates $Al_2(OH)_6$. Notice the character of the final precipitate and compare it with the precipitate, due to impurities in the NaOH, which is usually obtained in the experiments with chromium.

Iron in Ferric Salts.—Use a solution of Fe_2Cl_6. Add NH_4Cl and NH_4OH. The precipitate is $Fe_2(OH)_6$. Add $(NH_4)_2S$. This changes the precipitate to FeS. Warm, filter, wash, dissolve in dil. HCl, boil the solution of $FeCl_2$ to expel H_2S, add a few drops of dil. HNO_3 and boil again. The $FeCl_2$ is oxidized to Fe_2Cl_6. Test a drop of the solution with KCNS. The compound formed is $Fe_2(CNS)_6$. Test another drop with $K_4FeC_6N_6$. The precipitate formed is ferric ferrocyanide, $Fe_4(FeC_6N_6)_3$, or "Prussian blue." *Nearly* neutralize the rest of the solution with Na_2CO_3, add $BaCO_3$, shake, filter and test the filtrate for iron with $K_4FeC_6N_6$. The $BaCO_3$ precipitates the iron as a basic salt.

Iron in Ferrous Salts.—Use a solution of $FeSO_4$ freshly prepared by dissolving an iron nail in dil. H_2SO_4. Test different portions of the solution with KCNS, $K_4FeC_6N_6$, $K_3FeC_6N_6$, and NaOH. Oxidize a portion of the $FeSO_4$ to ferric sulphate by boiling with HNO_3 and apply the same tests. Ferric salts are reduced to ferrous salts by H_2S. Prove it, using the knowledge gained from tests above. Write all of the reactions.

Cobalt.—Use a solution of $Co(NO_3)_2$. Add NH_4Cl and NH_4OH, then $(NH_4)_2S$, warm, filter and test the precipitate with the borax bead in the oxidizing flame. Pour over the precipitate on the filter* a little water and cold dil. HCl and test the filtrate for Co with NH_4OH and $(NH_4)_2S$. A *slight* precipitate will usually be obtained. Transfer the remainder of the CoS to a porcelain dish, dissolve in a little boiling dil. aqua regia, and evaporate the solution of $CoCl_2$ to a few drops. Rinse back into a test tube with a *very little* water so that the volume is less than 1 cc. Add NaOH drop by drop till a slight permanent precipitate, $Co(OH)_2$, remains after shaking. Dissolve this in a few drops of acetic acid. The volume should not now be more than 2 cc. Add an equal volume of KNO_2, warm gently and allow to stand for twelve hours. The yellow precipitate is $Co_2(NO_2)_6.(KNO_2)_6 + nH_2O$. Filter, add NaOH to the filtrate and test a precipitate, if one is obtained, with the borax bead.

Nickel.—Use a solution of $Ni(NO_3)_2$. Try the same experiments as with cobalt. Notice that the first filtrate from the NiS is dark. NiS can be precipitated from it by adding dil. HCl.

Mix five parts of a nickel solution with one part of a cobalt solution, precipitate with $(NH_4)_2S$ and test the precipitate with the borax bead. Is the color obtained characteristic of cobalt or of nickel?

Manganese.—Use a solution of $MnSO_4$. Add NH_4Cl, NH_4OH, then $(NH_4)_2S$, warm, filter, wash, dissolve the MnS in cold dil. HCl, boil to expel H_2S, *nearly* neutralize with Na_2CO_3, add $BaCO_3$ to the cold solution, shake and filter. To the filtrate add two or three drops of dil. HCl, boil to expel CO_2, add NH_4OH and $(NH_4)_2S$, warm, filter, dissolve the MnS in dil. HCl, boil to expel H_2S, add NaOH and filter. Test the filtrate with H_2S and the precipitate, $Mn(OH)_2$, with a bead of Na_2CO_3 in the oxidizing flame. The green color of the bead is caused by sodium manganate, Na_2MnO_4.

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*Precipitated sulphides should always be further treated as soon as possible, as they are liable to be oxidized to sulphates by the air.

Zinc.—Use a solution of $ZnSO_4$. Try the same experiments as with manganese. Heat the last precipitate of ZnS on charcoal, moisten with $Co(NO_3)_2$ and heat again.

ANALYSIS.

III.—Ammonium Sulphide Group.

(16). **Precipitation** —Put a *small portion* of the solution into a test tube, boil to expel H_2S if present, add 2 drops of dil. HNO_3 and boil again. If the solution becomes yellow, *iron* is probably present. Add NH_4Cl, then NH_4OH just to alkaline reaction and warm.

a. No precipitate is formed. Absence of Al, Cr, and Fe.* Add $(NH_4)_2S$ and warm.

aa. No precipitate is formed. Absence of Co, Ni, Zn and Mn. Pass to (25).

bb. A precipitate may contain CoS, NiS, ZnS and MnS. Add to the whole solution NH_4Cl, to prevent the precipitation of Ba, Sr, Ca or Mg, then NH_4OH just to alkaline reaction, and $(NH_4)_2S$. Warm, filter and wash the precipitate *at once* (otherwise the sulphides might be oxidized to sulphates by the air) with hot water to which a little $(NH_4)_2S$ has been added. Treat the filtrate as in b. Put the precipitate into a porcelain dish with a little water and cold

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*If the solution contains tartaric acid or other organic matter, this may prevent the precipitation of Al, Cr, and Fe by NH_4OH. In that case the solution must be evaporated to dryness and the residue fused on platinum with Na_2CO_3 and KNO_3. The fused mass, *after removal from the platinum*, is dissolved in dil. HCl, H_2S is passed through the solution to reduce chromic acid and it is then treated as above.

Cobalt.—[illegible] Co(NO$_3$)$_2$. Add NH$_4$Cl and [illegible] the precipitate [illegible]. Pour over the [illegible] dil. HCl and [illegible] NH$_4$OH and (NH$_4$)$_2$S. A *slight* [illegible]. Transfer the remainder [illegible] in a little boiling [illegible] CoCl$_2$ to a few [illegible] with a *very little* water [illegible]. Add NaOH drop by [illegible] Co(OH)$_2$ remains [illegible] drops of acetic acid. [illegible] than 2 cc. Add an [illegible] KNO$_2$ [illegible] and allow to stand for [illegible]. The [illegible] is Co$_2$(NO$_2$)$_6$.6KNO$_2$ [illegible] (NH$_4$)$_2$S to the filtrate and test a precip[illegible] with the borax bead.

Nickel.—[illegible] Ni(NO$_3$)$_2$. Try the same ex[illegible] that the first filtrate from [illegible] NiS can be precipitated from it by adding [illegible] HCl.

Mix two parts of a nickel solution with one part of a cobalt solution, precipitate with (NH$_4$)$_2$S and test the precipitate with the borax bead. Is the color obtained characteristic of cobalt or of nickel?

Manganese.—Use a solution of MnSO$_4$. Add NH$_4$Cl, NH$_4$OH, then (NH$_4$)$_2$S, warm, filter, wash, dissolve the MnS in cold dil. HCl, boil to expel H$_2$S, *nearly* neutralize with Na$_2$CO$_3$, add BaCO$_3$ to the cold solution, shake and filter. To the filtrate add two or three drops of dil. HCl, boil to expel CO$_2$, add NH$_4$OH and (NH$_4$)$_2$S, warm, filter, dissolve the MnS in dil. HCl, boil to expel H$_2$S, add NaOH and filter. Test the filtrate with H$_2$S and the precipitate, Mn(OH)$_2$, with a bead of Na_2CO_3 in the oxidizing flame. The green color of the bead is caused by sodium manganate, Na_2MnO_4.

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*Precipitated sulphides should always be further treated as soon as possible, as they are liable to be oxidized to sulphates

Zinc.—Use a solution of $ZnSO_4$. Try the same experiments as with manganese. Heat the last precipitate of ZnS on charcoal, moisten with $Co(NO_3)_2$ and heat again.

ANALYSIS.

III.—Ammonium Sulphide Group.

(16). **Precipitation**.—Put a *small portion* of the solution into a test tube, boil to expel H_2S if present, add 2 drops of dil. HNO_3 and boil again. If the solution becomes yellow, *iron* is probably present. Add NH_4Cl, then NH_4OH just to alkaline reaction and warm.

- a. No precipitate is formed. Absence of Al, Cr, and Fe.* Add $(NH_4)_2S$ and warm.
  - aa. No precipitate is formed. Absence of Co, Ni, Zn and Mn. Pass to (25).
  - bb. A precipitate may contain CoS, NiS, ZnS and MnS. Add to the whole solution NH_4Cl, to prevent the precipitation of Ba, Sr, Ca or Mg, then NH_4OH just to alkaline reaction, and $(NH_4)_2S$. Warm, filter and wash the precipitate *at once* (otherwise the sulphides might be oxidized t[illegible]lphates by the air) with hot water to whic[illegible] $(NH_4)_2S$ has been added. Treat the [illegible] in b. Put the precipitate into a po[illegible] little water and cold

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olut[illegible] other organic matter, this
ent[illegible] and Fe by NH_4OH. In
the[illegible] to dryness and the residue
pla[illegible] O_3. The fused mass, *after*
fro[illegible] in dil. HCl, H_2S is passed
th[illegible] cid and it is then treated as

dil. HCl. Digest for a short time, filter and wash. The precipitate may contain CoS and NiS. Treat as in (17). The filtrate may contain $MnCl_2$ and $ZnCl_2$. Boil to expel H_2S, add NaOH in excess to the cold solution and filter, if a precipitate of $Mn(OH)_2$ is formed. Test the precipitate with a Na_2CO_3 bead in the O. F. A green color is caused by sodium manganate, Na_2MnO_4. Test the NaOH solution or filtrate with H_2S. A white precipitate is ZnS.

b. A precipitate is formed on adding NH_4OH.* This will be reddish, if $Fe_2(OH)_6$; green, if $Cr_2(OH)_6$; white and flocculent, if $Al_2(OH)_6$; or white, if a phosphate or similar compound of Ba, Sr, Ca, or Mg. Add to the whole solution, NH_4Cl, then NH_4OH just to alkaline reaction, and $(NH_4)_2S$. Warm, filter and wash at once (to prevent oxidation) with hot water to which a little $(NH_4)_2S$ has been added. Treat the precipitate by (17). Test the filtrate with a drop of $(NH_4)_2S$ to make sure the metals of the group have been completely precipitated. If the filtrate is dark colored, the presence of Ni is indicated. In that case add dil. HCl just to acid reaction, filter cold, and to the filtrate add at once NH_4OH to alkaline reaction and reserve it to test by (25). Test the precipitate for Ni with a borax bead in the O. F. If necessary, wrap the portion of the filter to which the precipitate adheres around the bead and burn, heating in the O. F. till the bead is clear. The bead should be violet when hot and reddish brown when cold. If Ni can be found here, the tedious separation of **(17)** will be unnecessary.

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*The following shorter method gives fairly satisfactory results in the absence of chromium and of phosphates: After boiling with HNO_3 to oxidize the iron, add NH_4Cl and NH_4OH and filter at once. Test the filtrate for Co, Ni, Mn and Zn, as in a. Wash the precipitate, dissolve a little of it in dil. HCl and test with KCNS for iron. Boil the remainder of the precipitate with NaOH in a porcelain dish, filter, add HCl to acid reaction, then NH_4OH to alkaline reaction and warm. A precipitate is $Al_2(OH)_6$.

(17). Cobalt and Nickel.—The precipitate may contain $Al_2(OH)_6$, $Cr_2(OH)_6$, FeS, CoS, NiS, MnS, ZnS, and also phosphates or similar compounds of Mg, Ca, Sr or Ba. Put it in a porcelain dish with a little water and add cold dil. HCl. Digest for a short time, filter and wash. Treat the filtrate, which contains everything but Co and Ni, by **(18)** and **(19)**. Test the precipitate with a borax bead in the O. F. If the bead is violet when hot and reddish brown when cold, Ni is present and Co is absent, or at least only a trace is present. If blue, Co is present. In that case put the precipitate in a porcelain dish, dissolve in a little dilute boiling aqua regia and evaporate the solution so far that on removing the lamp the residue will become almost dry. Transfer to a test tube with a few drops of water,* (1 cc or less), add NaOH drop by drop till a slight permanent precipitate is formed, dissolve that in a little acetic acid. The volume should not now exceed 2 or 3 cc. Add an equal volume of KNO_2. Warm gently and allow to stand for 12 hours. A yellow precipitate is $Co_2(NO_2)_6(KNO_2)_6+nH_2O$. Filter, add NaOH to the filtrate and test a green precipitate of $Ni(OH)_2$ with the borax bead. The presence of Ni should not be considered certain till a characteristic nickel bead is obtained.

(18). Barium, Strontium and Calcium.—This paragraph can be omitted if phosphoric, silicic, boric, oxalic, hydrofluoric and tartaric acids can be proved to be absent, or if the original solution contains no free acid. (See page 59, a.) Otherwise, to *one-fourth* of the filtrate add dil. H_2SO_4 and boil. A precipitate may consist of $BaSO_4$ or $SrSO_4$, or, possibly of $CaSO_4$. Filter and wash. To the filtrate, or to the solution in which H_2SO_4 produces no precipitate, add 3 volumes of alcohol. A precipitate is $CaSO_4$. Filter, dissolve in water and add $(NH_2)_2C_2O_4$. A precipitate is CaC_2O_4. If a precipitate of $BaSO_4$ or of $SrSO_4$, or both, was obtained, fuse it on platinum foil with four parts of K_2CO_3 and Na_2CO_3. Boil the fused mass with water, filter and wash the $BaCO_3$ and $SrCO_3$ *thoroughly* with hot water. Then dissolve in acetic acid and test the solution as in (**27**).

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*This solution may be tested for Ni as follows: Add KCN till the precipitate first formed redissolves, then a drop of acetic acid and boil. Add now NaClO or bromine water and boil again. A black precipitate is nickelic hydroxide, $Ni_2(OH)_6$.

(**19**). **Iron.**—Boil the other three-fourths of the filtrate from the CoS and NiS in a porcelain dish to expel H_2S. Add a few drops of dil. HNO_3 to oxidize $FeCl_2$ to Fe_2Cl_6 and evaporate nearly to dryness. Put a drop of KCNS on a watch glass and add to it by means of a clean glass rod, a drop of the solution. A red color is caused by $Fe_2(CNS)_6$. Whether the iron was originally present as a ferrous or a ferric salt must be determined by testing the original solution in HCl with KCNS and with $K_3FeC_6N_6$.

(**20**). **Aluminum.**—To the remainder of the solution add, if necessary, enough Fe_2Cl_6 to combine with the phosphoric acid present, so that a drop of the solution will give a yellowish and not a white precipitate when mixed with NH_4OH in a watch glass. *Nearly* neutralize the free acid with Na_2CO_3. (If an excess is accidentally added, dissolve the precipitate formed in a few drops of dil. HCl). Add $BaCO_3$ to the cold solution, shake repeatedly, and allow to stand until the solution above the precipitate is colorless. Filter and wash. The filtrate may contain $MnCl_2$, $ZnCl_2$ and $MgCl_2$, and always contains $BaCl_2$ Treat it by (**22**). The precipitate may contain Al, Cr and Fe as basic salts. Boil it in a porcelain dish with NaOH, filter and treat the precipitate, which will contain the chromium and iron, by **(21)**. To the filtrate add dil. HCl to acid reaction, then NH_4OH to alkaline reaction and warm. A white flocculent precipitate should be $Al_2((OH)_6$. However, the NaOH is rarely so pure but a precipitate will be obtained, even in the absence of Al. An equal quantity of the NaOH should, therefore, be acidified with dil. HCl and the solution treated with NH_4OH as above. If a precipitate is obtained, it will usually be possible, by comparing the character and amount of the two precipitates, to determine whether Al was present or not.

(21). Chromium —Fuse the precipitate on platinum foil with two parts of Na_2CO_3 and two parts of $KClO_3$. Boil the fused mass with a little water and filter. If the filtrate is yellow, K_2CrO_4 is present. Whether the chromium was present as an acid or a base may be known by the color of the original solution.

(22). Manganese.—To the filtrate containing $MnCl_2$ and $ZnCl_2$ add a drop of dil. HCl and boil in a porcelain dish to expel CO_2. Add NH_4OH and $(NH_4)_2S$. A precipitate may contain ZnS and MnS. Filter and wash. Treat the filtrate by **(24)**. Dissolve the precipitate in cold dil. HCl (a residue of CoS or NiS may be treated by **(17)**. Boil till H_2S is *completely* expelled, cool and add NaOH in excess. A precipitate may contain $Mn(OH)_2$. Filter and treat the filtrate by **(23)**. Test the precipitate with a Na_2CO_3 bead in the O. F. If a green bead is obtained it contains sodium manganate, Na_2MnO_4.

(23). Zinc.—Pass H_2S through the solution in NaOH obtained in **(22)**. A white precipitate is ZnS.

(24). Magnesium.—Add to the filtrate from the MnS and ZnS, dil. H_2SO_4 and boil to precipitate the Ba. Filter, and to the filtrate add NH_4OH and $(NH_4)_2C_2O_4$. If a precipitate of $Ca(C_2O_4)$ forms, filter and test the filtrate for Mg with Na_2HPO_4. See **(28)** for the properties of the precipitate.

GROUP IV.

Ammonium Carbonate Group.

To this group belong **Barium, Strontium**, and **Calcium.** The carbonates of the metals of the group are insoluble in NH_4OH in the presence of NH_4Cl. **Magnesium** carbonate is also insoluble in water but is soluble in NH_4Cl.

The hydroxides of the metals of the group are soluble with difficulty in pure water or in NaOH or KOH, ($Mg(OH)_2$ is almost insoluble), but are quite readily soluble in the presence of ammonium salts.

PRELIMINARY EXPERIMENTS.

Barium.—Use a solution of $BaCl_2$. Add $(NH_4)_2CO_3$, warm, filter, dissolve in $C_2H_4O_2$ and to a small portion of the solution add $CaSO_4$. Notice whether the precipitate of $BaSO_4$ is formed immediately or slowly. To the remainder of the solution add $K_2Cr_2O_7$. The precipitate is $BaCrO_4$. Filter and test the filtrate for Ba with NH_4OH and $(NH_4)_2CO_3$. Dissolve the $BaCrO_4$ in dil. HCl and add dil. H_2SO_4.

Strontium.—Use a solution of $SrCl_2$. Add $(NH_4)_2CO_3$, warm, filter, dissolve in $C_2H_4O_2$ and add $CaSO_4$ to a small portion of the solution. Warm, if necessary, and notice the difference between the conduct of Ba and Sr. To the remainder of the solution add $K_2Cr_2O_7$, then NH_4OH and $(NH_4)_2CO_3$, filter and wash. Dissolve the $SrCO_3$ in $C_2H_4O_2$ and test a small portion of the solution with $CaSO_4$ and warming. To the remainder add $(NH_4)_2SO_4$, boil for some time, filter and test the filtrate with NH_4OH and $(NH_4)_2C_2O_4$. If the $SrCl_2$ used was free from Ca, no precipitate should be obtained.

Calcium.—Use a solution of $CaCl_2$. Try the same experiments as with strontium. The last precipitate is calcium oxalate, CaC_2O_4.

If a spectroscope is available, study carefully the spectrum of each metal of the group.

ANALYSIS.

IV.—Ammonium Carbonate Group.

(25).—To a **small portion** of the solution add NH_4Cl, if no ammonium salt is present, then NH_4OH and $(NH_4)_2CO_3$ and warm.

a. No precipitate forms. Absence of more than traces of Ba, Sr, or Ca. Divide in two portions. To one add a drop of $(NH_4)_2SO_4$ and warm. A precipitate is $BaSO_4$. To the other add $(NH_4)_2C_2O_4$ and warm. A precipitate is CaC_2O_4. If either is formed, treat the main solution with the same reagent, warm, filter and treat the filtrate by (28).

b. A precipitate may contain $BaCO_3$, $SrCO_3$ or $CaCO_3$. Treat the whole solution in the same manner as the sample, warm, filter and wash. Remove traces of Ba and Ca from the filtrate by adding a few drops of $(NH_4)_2SO_4$ and $(NH_4)_2C_2O_4$, warming and filtering. Treat the filtrate by (28). Dissolve the carbonates on the filter with acetic acid, and test the solution as follows:

(26). To one-fourth of the solution add a few drops of dil. HCl and evaporate to dryness in a porcelain dish. Dissolve in a drop of water and test with the spectorscope using a *clean* platinum wire.

(27). Add to a small portion of the solution in acetic acid, $CaSO_4$.

a. *No precipitate forms even after some time and warming.* **Ba** and **Sr** are absent. Confirm the presence of **Ca** by adding to the remainder of the solution, $(NH_4)_2C_2O_4$. A white pulverulent precipitate is CaC_2O_4.

b. *A precipitate forms immediately.* **Ba** is present. Add to the remainder of the solution $K_2Cr_2O_7$ and filter. The filtrate must be yellow. Add to it

NH_4OH and $(NH_4)_2CO_3$. A precipitate may contain $SrCO_3$ and $CaCO_3$. Warm, filter, wash and dissolve in $C_2H_4O_2$. To a small portion of the solution add $CaSO_4$ and boil. If a precipitate of $SrSO_4$ forms, treat the remainder of the solution as in c. If Sr is absent, test the remainder of the solution for Ca with NH_4OH and $(NH_4)_2C_2O_4$.

c. *A precipitate forms slowly or after warming.* **Ba** is absent. **Sr** is present. Add to the remainder of the solution $(NH_4)_2SO_4$ and boil for some time. Filter and test the filtrate with NH_4OH and $(NH_4)_2C_2O_4$. A precipitate is CaC_2O_4.

GROUP V.

Soluble Group.

To this group belong **Magnesium, Sodium, Potassium** and **Ammonium.** The chlorides, hydroxides, sulphides and carbonates of the metals of this group are soluble in NH_4Cl in the presence of ammoninum salts. There is no common precipitant for the group and very few insoluble salts of the last three metals are known.

PRELIMINARY EXPERIMENTS.

Magnesium.—Use a solution of $MgCl_2$ or $Mg(NO_3)_2$. Add to a small portion, NH_4OH, NH_4Cl and Na_2HPO_4. The precipitate is $MgNH_4PO_4$. To another portion add $Ba(OH)_2$. The precipitate is $Mg(OH)_2$.

Sodium.—Use a solution of NaCl. Test in the flame. Add one drop of a solution of NaCl to ten drops of a solution of KCl and test in the flame.

Potassium.—Use a solution of KCl. Test in the flame using a *clean* platinum wire. To a few drops of the solution in a watch glass add a few drops of a solution of $PtCl_4$, (the solution really contains chloro-platinic acid H_2PtCl_6), evaporate to dryness on a water bath and take up with a little water. The yellow crystalline precipitate is K_2PtCl_6, potassium chloro-platinate.

Ammonium.—Use a solution of NH_4Cl. Test a few drops with $PtCl_4$ as directed under potassium. The precipitate is $(NH_4)_2PtCl_6$. Evaporate a little of the NH_4Cl solution to dryness in a porcelain dish and heat the residue. Warm another portion with NaOH in a test tube. Notice the odor, the reaction of the gas given off, and the fumes when a rod dipped in conc. HCl is held at the mouth of the tube. The gas is NH_3.

ANALYSIS.

V.—Soluble Group.

(28). **Magnesium.**—If the solution has become dilute by the addition of reagents, concentrate it by evaporation in a porcelain dish and filter, if necessary. To a *small portion* of the solution add NH_4Cl, NH_4OH and Na_2HPO_4. If no precipitate forms at once, shake vigorously and rub the inside wall of the test tube to aid in the formation of the white chrystaline precipitate of $MgNH_4PO_4$. A white flocculent precipitate of $AlPO_4$ is sometimes obtained, but if this is filtered off, it will be found to be insoluble in $C_2H_4O_2$, while the $MgNH_4PO_4$ is soluble and may be reprecipitated from the solution by NH_4OH.

(29). **Sodium.**—Evaporate the remainder of the solution to dryness in a small porcelain dish and ignite on a wire gauze or a triangle till ammonium salts are *completely* expelled. Dissolve the residue in a few drops of water. Dip in the solution a platinum wire which has been carefully cleaned and held in the flame till it no longer imparts to it any color, and test in the flame. If a violet flame is obtained K is present and Na is absent. If the flame is yellow, Na is present, but the test is so delicate that unless the flame is brilliant it is probably an accidental impurity. If Na is found, or if the results of the flame test are unsatisfactory, test the solution for K by (**30**), if Mg is absent. If Mg is present, add $Ba(OH)_2$ to alkaline reaction, filter, add NH_4OH and $(NH_4)_2CO_3$, warm, filter from the $BaCO_3$, evaporate the filtrate to dryness, ignite on wire gauze till ammonium salts are *completely* expelled, and test by (**30**).

(**30**). **Potassium.**—Dissolve the residue in a few drops of water, filter, if necessary, through a small filter into a watch glass, add a few drops of $PtCl_4$ and evaporate to dryness on a water bath. Dissolve the residue in a *little* water and gather any precipitate which remains undissolved by tipping the glass slightly in different directions. A heavy, yellow crystalline precipitate is K_2PtCl_6. If hydriodic acid is present, a dark red solution will be obtained on taking up with water, but if a few drops of aqua regia are added and the solution evaporated again, the difficulty will be removed.

(**31**). **Ammonium.**—Test the original substance for NH_4 by putting a portion of it in a beaker and adding NaOH to alkaline reaction. Cover the beaker with a watch glass, on the lower side of which is placed a moistened slip of red litmus paper, and warm gently but do not boil. If the litmus turns blue it is caused by NH_3. If much ammonium is present, the odor of NH_3 will be perceptible.

TABLES OF REACTIONS.

REACTIONS OF THE METALS.

Group I.

Reagent.	***Silver.*** ($AgNO_3$.)	***Mercury (ous).*** ($HgNO_3$).	***Lead.*** $Pb(NO_3)_2$.
Hydrochloric Acid. HCl.	Silver Chloride. $AgCl$. White pr., insol. in acids, soluble in NH_4OH, in KCN, in $Na_2S_2O_3$. Darkens in the light.	Mercurous Chloride. $HgCl$. White pr. Sol. in HNO_3, in aqua regia. Converted to Hg_2NH_2Cl by NH_4OH.	Lead Chloride. $PbCl_2$. White pr., slightly soluble in cold water, more easily in hot water. Converted to an insoluble basic salt by NH_4OH.
Hydrogen Sulphide. H_2S.	Silver Sulphide. Ag_2S. Black pr., insol. in $(NH_4)_2S$. Sol. in hot HNO_3.	Mercuric Sulphide and Mercury. $HgS+Hg$. Black pr., partly sol. in HNO_3. Sol. in aqua regia.	Lead Sulphide. PbS. Black pr., insol. in $(NH_4)_2S$. Sol. in HNO_3.
Sulphuric Acid. H_2SO_4.	Silver Sulphate. Ag_2SO_4. White precip., only in concentrated solutions.	Mercurous Sulphate. Hg_2SO_4. White pr., slightly sol. in water.	Lead Sulphate. $PbSO_4$. White pr., insol. in excess, slightly sol. HNO_3. Soluble in $(NH_4)_2C_4H_4O_6$.
Sodium Hydroxide. $NaOH$.	Silver Oxide. Ag_2O. Brown pr., sol. in HNO_3. in NH_4OH.	Mercurous Oxide. Hg_2O. Black pr., soluble in HNO_3, insol. in NH_4OH.	Lead Hydroxide. $Pb(OH)_2$. White pr. Sol. in excess, in HNO_3, insol. in NH_4OH.
Ammonium Hydroxide. NH_4OH	Silver Oxide. Ag_2O. Brown precip., soluble in excess.	Mercurous amido Salt. Black precip., insol. in excess.	Basic Salt. White pr., insol. in excess.
Sodium Carbonate. Na_2CO_3.	Silver Carbonate. Ag_2CO_3. White precip., soluble in NH_4OH.	Basic Salt. Yellow pr., quickly becoming black.	Basic Lead Carbonate. $2PbCO_3Pb(OH)_2$. White pr., "White Lead."
Potassium Chromate. K_2CrO_4.	Silver Chromate. Ag_2CrO_4. Dark red precip., soluble in HNO_3, in NH_4OH.	Basic Chromate. Dark red pr., slightly sol. in HNO_3.	Lead Chromate. $PbCrO_4$. Yellow pr. Slightly sol. in HNO_3. Sol. in $NaOH$.
Potassium Ferrocyanide. $K_4FeC_6N_6$.	Silver Ferrocyanide. $Ag_4FeC_6N_6$. White precip., insoluble in NH_4OH.	Mercurous Ferrocyanide. $Hg_4FeC_6N_6$. White precipitate.	Lead Ferrocyanide. $Pb_2FeC_6N_6$. White precipitate.
Stannous Chloride. $SnCl_2$.	Silver Chloride. $AgCl$. White precip.	Mercury. Hg. Dark grey precip.	Lead Chloride. $PbCl_2$. White precipitate.

Group II—Sub-Group A.

REAGENT.	*Mercury (ic).* $HgCl_2$	*Copper.* $CuSO_4$.	*Bismuth.* $BiCl_3$.	*Cadmium.* $CdSO_4$.
Hydrogen Sulphide. H_2S.	Mercuric Sulphide HgS. First white, then yellow, reddish brown and black pr., insol. in HNO_3. in $(NH_4)_2S$. Sol. in aq regia, in $Na_2S+NaOH$.	Copper Sulphide CuS. Black pr., sol. in HNO_3, in KCN. Very slightly sol. in $(NH_4)_2S$. Insol. in hot dil. H_2SO_4.	Bismuth Sulphide. Bi_2S_3. Dark brown pr. sol. in HNO_3, insol. in KCN or in $(NH_4)_2S$	Cadmium Sulphide. CdS. Yellow pr. sol. in HNO_3, in hot diluted H_2SO_4, insol. in KCN, in $(NH_4)_2S$.
Sulphuric Acid. H_2SO_4.	No precipitate.	No precipitate.	Sometimes a pr. forms on long standing.	No precipitate.
Ammonium Hydroxide. NH_4OH.	Mercuric Amido Chloride. $HgNH_2Cl$ White precip.	Basic Copper ammonium salt. Greenish blue pr. sol. in excess to a blue solution	Bismuthyl Hydroxide. $BiO\ OH$. White pr., insol. in excess.	Cadmium Hydroxide. $Cd(OH)_2$. White precip., sol. in excess.
Sodium Hydroxide. $NaOH$.	Mercuric Oxide. HgO. Yellow pr. In presence of NH_4Cl, white pr. of $HgNH_2Cl$	Copper Hydroxide. $Cu(OH)_2$. Blue pr. changed to black CuO by boiling.	Bismuthyl Hydroxide. $BiO\ OH$. White precip.	Cadmium Hydroxide. $Cd(OH)_2$. White precip.
Sodium Carbonate. Na_2CO_3.	Mercuric Oxychloride. $HgCl_2+nHgO$. Reddish br. pr., changed to yellow HgO by boiling.	Rasic Copper Carbonate. Blue pr. changed to black CuO by boiling.	Bismuthyl Carbonate. $(BiO)_2CO_3$. White precip.	Cadmium Carbonate. $CdCO_3$. White precip.
Potassium Chromate. K_2CrO_4.	Mercuric Chromate. $HgCrO_4$. Reddish yellow pr., soluble in HNO_3.	Copper Chromate. $CuCrO_4$. Reddish brown precip., sol in NH_4OH to a green solution	Bismuth Chromate. $Bi_2(CrO_4)_3$. Yellow pr., sol. in HNO_3, insol. in KOH.	——
Potassium Ferrocyanide. $K_4FeC_6N_6$.	——	Copper Ferrocyanide. $Cu_2FeC_6N_6$. Reddish brown pr. slightly sol. in NH_4OH, in sol. in acids.	Bismuth Ferrocyanide. White pr., insol. in HCl.	Cadmium Ferrocyanide. $Cd_2FeC_6N_6$. Yellowish white precip., sol. in HCl.
Stannous Chloride. $SnCl_2$.	Mercurous Chloride. $HgCl$. White pr. An excess of $SnCl_2$ gives gray Hg.	Cuprous Chloride. Cu_2Cl_2. White precip., only in conc. solutions.	Darkens a pr. of $BiOOH$, changing it to Bi_2O_2.	——
Water. H_2O.	——	——	Basic Bismuth Salt. $BiOCl$. White pr., sol. in HCl.	——

Reagent.	***Arsenic.***		***Antimony.***	
	Arsenious Salts. K_3AsO_3.	**Arsenic Salts.** KH_2AsO_4.	**Antimonious Salts.** $SbCl_3$.	**Antimonic Salts.** $KSbO_3$
Hydrogen Sulphide. H_2S.	Arsenic trisulphide. As_2S_3. Yellow pr., sol. in alkalies and alkali sulphides. Insol. in conc. HCl.	Arsenic trisulphide and sulphur. As_2S_3+2S. Yellow precip. formi'g slowly and on warming.	Antimony trisulphide. Sb_2S_3. Orange pr. sol. in conc. HCl, in Alkalies and alkali sulphides.	Antimony pentasulphide Sb_2S_5. Orange pr. sol. in conc. HCl, in alkalies and alkali sulphides.
Ammonium Sulphide. $(NH_4)_2S$.	Arsenic trisulphide. As_2S_3. Only in acid solutions. Sol. in excess.	Arsenic pentasulphide As_2S_5. Only in acid solutions. Sol. in excess.	Antimony trisulphide, Sb_2S_3. Sol. in excess.	Antimony pentasulphide Sb_2S_5. Sol. in excess.
Sodium Hydroxide. NaOH.	——	——	Antimonious Hydroxide. $Sb(OH)_3$. White pr., sol. in excess.	Sodium Metantimonate. $NaSbO_3$. White pr. sol. in excess.
Potassium Hydroxide. KOH.	——	——	Antimonius Hydroxide. $Sb(OH)_3$	——
Ammonium Hydroxide. NH_4OH.	——	——	Antimonious Hydroxide. $Sb(OH)_3$. Insol. in excess.	Ammonium Metantimonate. NH_4SbO_3. Nearly insol. in excess.
Sodium Carbonate. Na_2CO_3.	——	——	Antimonious Hydroxide. $Sb(OH)_3$. Sol. in excess on boiling.	——
Silver Nitrate. $AgNO_3$.	Silver Arsenite. Ag_3AsO_3. Yellow pr., sol. in HNO_3, in. NH_4OH.	Silver Arsenate Ag_3AsO_4 Reddish brown pr., sol. in HNO_3, in NH_4OH.	Silver Chloride and Antimony trioxide. $AgCl+Sb_2O_3$ White precip.	Silver Antimonate. Ag_3SbO_4. White pr., sol. in NH_4OH.
Copper Sulphate. $CuSO_4$.	Copper Arsenite. $CuHAsO_3$. Yellowish green pr. sol. in HNO_3, in NH_4OH, in NaOH.	Copper Arsenate. $CuHAsO_4$. Greenish blue pr. soluble in HNO_3, in NH_4OH.	Antimony Oxychloride. SbOCl. White pr. caused by dilution.	Copper Antimonate. Brown precip.
Mercuric Chloride. $HgCl_2$.	Mercuric Arsenite. $Hg_3(AsO_3)_2$. White pr., sol. in acids.	——	Antimony Oxychloride. Caused by dilution. Sol. in conc. HCl.	——
Stannous and Stannic Chlorides. $SnCl_2+SnCl_4$.	——	——	——	——
Magnesia Mixture. NH_4Cl, NH_4OH, $MgSO_4$	——	Magnesium Ammonium Arsenate. $MgNH_4AsO_4$. White crys. pr., sol. in $C_2H_4O_2$	Same pr. as by NH_4OH.	Same precip. as by NH_4OH.
Metallic Zinc and Hydrochloric Acid. (Marsh's test.)	Hydrogen and Hydrogen Arsenide. The flame deposits a spot on porcelain. Sol. in sodium hypochlorite. Sol. in $(NH_4)_2S$. On evaporation the yellow residue is insol. in conc. HCl.		Hydrogen and Hydrogen Antimonide. The flame deposits a spot on porcelain. Insol. in sodium hypochlorite. Sol. in $(NH_4)_2S$. On evaporation the orange residue is sol. in conc. HCl.	

Tin.		*Platinum.*	*Gold.*
STANNOUS SALTS. $SnCl_2$.	STANNIC SALTS. $SnCl_4$.	$PtCl_4$.	$AuCl_3$.
Stannous Sulphide. SnS. Dark brown pr., sol. in conc. HCl. in alkalies. Difficultly sol. in yellow $(NH_4)_2S$.	Stanic Sulphide. SnS_2. Yellow pr., sol. in conc. HCl. in alkalies and alkali sulphides, in alkali carbonates.	Platinic Sulphide. PtS_2. Dark brown pr., insol. in conc. HCl. Difficultly sol. in alkali sulphides. Sol. in aqua regia.	Gold Sulphide. Au_2S_3. Black pr. insol. in conc. HCl. Sol. in alkali sulphides, sol. in aqua regia.
Stannous Sulphide. SnS. Sol. in yellow $(NH_4)_2S$.	Stannic Sulphide. SnS_2. Sol. in excess.	Platinic Sulphide. PtS_2. Sol. in excess.	Gold Sulphide Au_2S_3. Sol. in excess.
Stannous Hydroxide $Sn(OH)_2$. White pr., sol. in excess.	Stannic Hydroxide. $Sn(OH)_4$. White pr., sol. in excess.	—	—
Stannous Hydroxide $Sn(OH)_2$.	Stannic Hydroxide. $Sn(OH)_4$.	Potassium Chloroplatinate. K_2PtCl_6. Yellow pr. in presence of free HCl.	—
Stannous Hydroxide $Sn(OH)_2$. Insol. in excess. Darkens on boiling.	Stannic Hydroxide. $Sn(OH)_4$. Slightly sol. in excess.	Ammonium Chloroplatinate. $(NH_4)_2PtCl_6$. Yellow pr., sol. in great excess.	Fulminating Gold. $Au_2O_3 2NH_3$. Yellow pr., insol. in excess.
Stannous Hydroxide $Sn(OH)_2$. Insol. in excess.	Stannic Hydroxide. $Sn(OH)_4$. Insol. in excess.	—	—
Silver Chloride and Silver. $AgCl+Ag$.	Silver Cloride. $AgCl$.	Silver Chloride and Platinum Oxide. $AgCl+PtO_2$. Brown precipitate.	Silver Chloride and Gold Oxide. $AgCl+Au_2O_3$. Brown precipitate.
Cuprous Chloride. Cu_2Cl_2. White pr., sol. in acids.	—	—	—
Mercurous Chloride. $HgCl$. White pr., insol. in cold conc. HCl.	—	—	—
—	—	—	Purple of Cassius. Purple red precip. or coloration.
Same precip. as by NH_4OH.	Same precip. as by NH_4OH.	Same precip. as by NH_4OH.	Same precip. as by NH_4OH.
Metallic Tin.	In neutral solution. also Stannic Hydroxide.	Platinum. Black precipitate.	Gold. Brown precipitate.

Reagent.	***Aluminium*** $Al_2K_2(SO_4)_4$.	***Chromium.*** $Cr_2K_2(SO_4)_4$	***Uranium*** $UO_2(NO_3)_2$.	***Ferric Salts.*** Fe_2Cl_6.
Ammonium Sulphide. $(NH_4)_2S$.	Aluminium Hydroxide. $Al_2(OH)_6$. White flocculent pr.	Chromium Hydroxide. $Cr_2(OH)_6$. Grayish green pr.	Uranyl Sulphide. UO_2S Dk. br. pr, somewhat sol. in exc. of colorless$(NH_4)_2S$.	Iron Sulphide FeS(+S). Black pr. Sol. in acids to a ferrous salt.
Hydrogen Sulphide. H_2S.	——	——	——	Reduced to a ferrous salt with separation of sulphur.
Ammonium Hydroxide. NH_4OH.	Aluminium Hydroxide. $Al_2(OH)_6$. White flocculent pr. very slighly sol. in excess.	Chromium Hydroxide. $Cr_2(OH)_6$. Grayish blue or green precip. slightly sol. in excess with a red color.	Ammonium Uranate. $(NH_4)_2U_2O_7$. Yellow pr.	Ferric Hydroxide. $Fe_2(OH)_6$ Reddish brown pr. insol. in excess.
Sodium Hydroxide. NaOH.	Aluminium Hydroxide. $Al_2(OH)_6$. White pr.sol.in excess as Na_3AlO_3.	Chromium Hydroxide. $Cr_2(OH)_6$. Sol. in exess to a green sol. reprecipitated by boiling.	Sodium Uranate. $Na_2U_2O_7$. Yellow pr. sol. in($NH_4)_2CO_3$.	Ferric Hydroxide. $Fe_2(OH)_6$ Reddish brown pr. insol. in excess.
Barium Carbonate. $BaCO_3$.	Aluminium Hydroxide. $Al_2(OH)_6$. White pr.	Basic Salt. Greenish pr.	Uranyl Hydroxide. $UO_2(OH)_2$ Yellow pr.	Basic Salt. Reddish brown pr.
Sodium Phosphate. Na_2HPO_4	Aluminium Phosphate. $AlPO_4$. White pr.sol.in NaOH. insol. in $C_2H_4O_2$.	Cromium Phosphate. $CrPO_4$. Green pr.	Uranly Phosphate. UO_2HPO_4 Yellowish white pr.	Ferric Phosphate. $FePO_4$. Yellowish white pr. sol. in excess. Insol in $C_2H_4O_2$.
Ammonium Carbonate. $(NH_4)_2CO_3$.	Aluminium Hydroxide. $Al_2(OH)_6$. White pr.	Cromium Hydroxide. $Cr_2(OH)_6$. Grayish green pr. sol. excess.	Ammonium Uranyl Carbonate $2(NH_4)_2CO_3 + UO_2CO_3$. Yellow pr. sol. in excess, reprecipitated by alkalies.	Basic Salt. Reddish brown pr.
Potassium Ferrocyanide $K_4FeC_6N_6$.	White pr.forms slowly.	——	Uranyl Potassium Ferrocyanide $K_4Fe_6N_6 + 2(UO_2)_2FeC_6N_6$. Reddish brown pr.	Ferric Ferrocyanide. $Fe_4(FeC_6N_6)_3$. Dark blue pr. insol. in HCl. Decomposed by NaOH.
Potassium Ferricyanide $K_3FeC_6N_6$.	——	——	——	Reddish brown color.
Potassium Sulphocyanate. KCNS.	——	——	Red color.	Blood red solution. $Fe_2(CNS)_6$. Most delicate with HCl.
Borax bead.	——	Yellowish green hot. Emerald green, cold.	Yellow in O. F. Green in R. F.	Yellow in O. F. Green in R. F.

Ferrous Salts, $FeSO_4$.	***Cobalt,*** $CoCl_2$.	***Nickel.*** $NiCl_2$.	***Manganese.*** $MnSO_4$.	***Zinc.*** $ZnSO_4$.
Iron Sulphide. $Fe S$. Black pr. sol. in HCl, in $C_2H_4O_2$. Oxidizes in the air.	Cobalt Sulphide. CoS. Black pr. insol. in $C_2H_4O_2$, almost insol. in HCl. Sol in aq. reg.	Nickel Sulphide. NiS. Black pr. slightly sol. in excess. Almost insol. in HCl. Sol. in aq. reg.	Manganese Sulphide. MnS Flesh col'd pr. sol. in HCl. in $C_2H_4O_2$. Oxidizes in the air.	Zinc Sulphide. ZnS. White pr. insol. in $C_2H_4O_2$. Sol. in HCl.
No pr. in acid solution.	Cobalt Sulphide. Only in neutral or alkaline solutions.	Nickel Sulphide. Only in neutral or alkaline solutions.	Manganese Sulphide Only in neutral or alkaline solutions.	Zinc Sulphide. Only in neutral alkaline or acetic acid solution.
Ferrous Hydroxide. $Fe(OH)_2$. White pr. becoming green and reddish brown. In presence of NH_4Cl, $Fe_2(OH)_6$ forms slowly.	Basic Salt. Blue pr. sol. in excess to a red sol. In presence of NH_4Cl, no pr.	Nickel Hydroxide. $Ni(OH)_2$. Green pr. sol. in excess to a blue sol. in presence of NH_4Cl no pr.	Manganese Hydroxide. $Mn(OH)_2$. White pr. becoming bro'n. In pres'nce of NH_4Cl a dark brown pr. forms slowly by oxidation	Zinc Hydroxide, $Zn(OH)_2$. White pr. sol. in excess. In presence of NH_4Cl no pr.
Ferrous Hydroxide. $Fe(OH)_2$. White pr. becoming green and reddish brown, insol. in excess.	Basic Salt. Blue pr. insol. in excess. Sol. in $C_2H_4O_2$, in NH_4OH.	Nickel Hydroxide. $Ni(OH)_2$. Apple green pr. insol. in excess. sol. in $C_2H_4O_2$. in NH_4OH.	Manganese Hydroxide. $Mn(OH)_2$. White pr. becoming bro'n. Insol. in excess.	Zinc Hydroxide $Zn(OH)_2$. White pr. sol. in excess. Re-precipitated by boiling.
No pr. in the cold except in the presence of a sulphate.	No pr. in the cold except in presence of a sulphate.	No pr. in the cold except in presence of a sulphate.	No pr. in the cold except in presence of a sulphate.	No pr. in the cold except in presence of a sulphate.
Ferrous Phosphate. $Fe_3(PO_4)_2$. White pr. becoming blue in the air.	Cobalt Phosphate. $Co_3(PO_4)_2$. Red pr. sol. in NH_4OH.	Nickel Phosphate. $Ni_3(PO_4)_2$. Green pr. sol. in NH_4OH.	Manganese Phosphate. $Mn_3(PO_4)_2$ White pr. sol. in NH_4OH.	Zinc Phosphate $Zn_3(PO_4)_2$. White pr. sol. in excess.
Ferrous Carbonate. $FeCO_3$. White pr. sol. in excess.	Basic Carbonate. Pink pr. sol. in excess.	Basic Carbonate. Green pr. sol. in excess.	Manganese Carbonate. $MnCO_3$. White pr. insol. in excess.	Basic Zinc Carbonate. White pr. sol. in excess.
Potassium Ferrous Ferrocyanide. $K_2Fe_2C_6N_6$. Bluish white pr. becoming blue	Cobalt Ferrocyanide. $Co_2FeC_6N_6$. Green pr. becoming greenish blue, insol in HCl.	Nickel Ferrocyanide $Ni_2FeC_6N_6$. Greenish white pr. insol. in HCl.	Manganese Ferrocyanide. $Mn_2FeC_6N_6$. Reddish white precip. sol. in HCl.	Zinc Ferrocyanide. $Zn_2FeC_6N_6$ White pr. sol. in HCl.
Ferrous Ferricyanide. $Fe_3(FeC_6N_6)_2$. Dark blue pr. insol. in HCl. Decomposed by NaOH.	Cobalt Ferricyanide. $Co_3(FeC_6N_6)_2$. Dark brown pr. insol. in HCl.	Nickel Ferricyanide. $Ni_3(FeC_6N_6)_2$. Yellowish green pr. insol. in HCl.	Manganese Ferricyanide. $Mn_3(FeC_6N_6)_2$. Brown pr insol. in HCl.	Zinc Ferricyanide. $Zn_3(FeC_6N_6)_2$. Yellowish br'n pr. sol. in HCl.
No color.	——	——	——	——
Yellow in O. F. Green in R. F.	Blue.	Violet, hot. Reddish brown, cold, gray and turbid in R. F.	Violet red, hot. Amethyst red, cold. Colorless in R. F.	——

Group IV.

Reagent.	***Barium.*** $BaCl_2$.	***Strontium.*** $SrCl_2$.	***Calcium.*** $CaCl_2$.	***Magnesium.*** $MgSO_4$.
Ammonium Carbonate. $(NH_4)_2CO_3$.	Barium Carbonate. $BaCO_3$. White pr. sol. in acids.	Strontium Carbonate. $SrCO_3$. White pr. sol. in acids.	Calcium Carbonate. $CaCO_3$. White pr. sol. in acids.	Basic Magnesium Carbonate. $MgCO_3 + nMg(OH)_2$. White pr. on warming and in absence of NH_4 salts.
Sodium Hydroxide. NaOH.	Barium Hydroxide. $Ba(OH)_2$. White pr. only in conc. solutions.	Strontium Hydroxide. $Sr(OH)_2$. White pr. difficultly sol. in water.	Calcium Hydroxide. $Ca(OH)_2$. White pr. difficultly sol. in water.	Magnesium Hydroxide. $Mg(OH)_2$. White pr. sol. in NH_4Cl.
Ammonium Hydroxide. NH_4OH.	No pr.	No pr.	No pr.	Magnesium Hydroxide. $Mg(OH)_2$. Sol. in NH_4Cl.
Sulphuric Acid. H_2SO_4.	Barium Sulphate. $BaSO_4$. White pr. insol. in water or in acids.	Strontium Sulphate. $SrSO_4$. White pr. very little sol. in water or in $(NH_4)_2SO_4$.	Calcium Sulphate. $CaSO_4$. White pr. somewhat sol. in water. Sol. in $(NH_4)_2SO_4$. Insol. in alcohol.	——
Ammonium Oxalate. $(NH_4)_2C_2O_4$.	Barium Oxalate. BaC_2O_4. White pr. sol. in HCl, slightly sol. in $C_2H_4O_2$.	Strontium Oxalate. SrC_2O_4. White pr. sol. in HCl, slightly sol. in $C_2H_4O_2$.	Calcium Oxalate. CaC_2O_4. White pr. sol. in HCl, almost insol. in $C_2H_4O_2$ or $H_2C_2O_4$.	No precipitate in dilute solutions.
Potassium Pyrochromate. $K_2Cr_2O_7$.	Barium Chromate. $BaCrO_4$. Yellow pr. insol. in $C_2H_4O_2$ in presence of $K_2Cr_2O_7$. Sol. in HCl.	——	——	——
Hydrofluosilicic Acid. H_2SiF_6.	Barium Fluosilicate. $BaSiF_6$. White pr. insol. in HCl.	——	——	——
Sodium Phosphate. Na_2HPO_4.	Barium Hydrogen Phosphate. $BaHPO_4$. White pr. sol. in acids.	Strontium Hydrogen Phosphate. $SrHPO_4$. White pr. sol. in acids.	Calcium Hydrogen Phosphate. $CaHPO_4$. White pr. sol. in acids.	Magnesium Hydrogen Phosphate. $MgHPO_4$. White pr. In presence of NH_4OH and NH_4Cl a white cryst. pr. of $MgNH_4PO_4$ is formed. Sol. in $C_2H_4O_2$.
Flame.	Yellowish green.	Bright red.	Yellowish red.	——

Group V.

	Lithium.	***Ammonium.***	***Sodium.***	***Potassium.***
Reagent.	LiCl.	NH_4Cl.	NaCl.	KCl.
Sodium Carbonate. Na_2CO_3.	Lithium Carbonate. Li_2CO_3. White pr. slightly sol. in water.	Ammonia. NH_3. On boiling.	——	——
Sodium Phosphate. Na_2HPO_4.	Lithium Phosphate. Li_3PO_4. White pr. slightly sol. in H_2O. Sol. in HCl.	——	——	——
Platinic Chloride $PtCl_4$. (H_2PtCl_6.)	——	Ammonium Chloroplatinate. $(NH_4)_2PtCl_6$. Yellow pr. slightly sol. in water. Insol. in alcohol.	——	Potassium Chloroplatinate. K_2PtCl_6. Yellow precip. slightly sol. in water, insol. in alcohol.
Tartaric Acid. $H_2C_4H_4O_6$	——	Mono-Ammonium Tartrate. $NH_4HC_4H_4O_6$ White cryst. pr. on shaking. Slightly sol. in water.	——	Mono-Potassium Tartrate. $KHC_4H_4O_6$. White cryst. pr. on shaking and rubbing. Somewhat sol. in water.
Potassium Pyroantimonate. $H_2K_2Sb_2O_7$.	——	——	Sodium Pyroantimonate. $Na_2H_2Sb_2O_7$. White cryst. pr., best in slightly alkaline solutions.	——
Nessler's Reagent. $HgI_2+KI+KOH$.	——	Reddish brown pr, or yellow color, very delicate.	——	——
Sodium Hydroxide. NaOH.	——	Evolves NH_3 on warming.	——	——
Hydrofluosilicic Acid. H_2SiF_6.	——	——	Sodium Fluosilicate. Na_2SiF_6. White pr. somewhat sol. in water.	Potassium Fluosilicate. K_2SiF_6. Transparent pr. slightly sol. in H_2O.
Flame.	Red.	——	Yellow.	Violet.

REACTION OF

I.—Inorganic

	Barium Chloride.	**Calcium Chloride.**	**Silver Nitrate.**
Sulphuric Acid. H_2SO_4.	White pr. insol. in acids.	White pr. somewhat sol. in water and in acids.	White pr. only in conc. solutions.
Hydrofluosilicic Acid. H_2SiF_6.	White pr. insol. in HCl.	——	——
Sulphurous Acid. H_2SO_3.	White pr. sol. in HCl.	White pr. sol. in HCl.	White pr. decomposed by boiling into Ag, Ag_2SO_4 and SO_2.
Thiosulphuric Acid, (Hyposulphurous.) $H_2S_2O_3$.	White pr. only in conc. solutions.	——	White pr. sol. in $Na_2S_2O_3$. Decomposed by boiling into H_2SO_4 and black Ag_2S.
Phosphoric Acid. H_3PO_4.	White pr. sol. in acids.	White pr. sol. in acids, even in $C_2H_4O_2$.	Yellow pr. sol. in HNO_3, in NH_4OH.
Pyrophosphoric Acid. $H_4P_2O_7$.	White pr. sol. in HCl.	White pr. sol. in an excess of the pyrophosphate.	White pr. sol. in HNO_3, in NH_4OH.
Metaphosphoric Acid. HPO_3.	White pr. sol. in. excess of the metaphosphate.	White pr.	White pr. sol. in HNO_3, in NH_4OH.
Arsenic Acid. H_3AsO_4.	White pr. sol. in acids.	White pr. sol. in $C_2H_4O_2$.	Reddish brown pr. sol. in HNO_3, in NH_4OH.
Arsenious Acid, H_3AsO_3.	White pr. sol. in acids.	White pr. sol. in $C_2H_4O_2$.	Yellow pr. sol. in HNO_3, in NH_4OH.
Chromic Acid. H_2CrO_4.	Yellow pr. sol. in HCl, in HNO_3, insol. in $C_2H_4O_2$.	——	Dark red pr. sol. in HNO_3.
Boric Acid. $H_3BO_3(H_2B_4O_7.)$	White pr. difficultly sol. in water, sol. in acids.	White pr. sol. in acids.	White pr. sol. in HNO_3, in NH_4OH.
Phosphorous Acid. H_3PO_3.	White pr. sol. in $C_2H_4O_2$.	White pr. sol. in NH_4Cl.	White pr. but deposits black Ag on warming.
Silicic Acid. H_2SiO_3.	White pr.	White pr.	Yellow pr. sol. in conc. solutions, sol. in HNO_3.
Carbonic Acid. H_2CO_3.	White pr. sol. in acids.	White pr.	Gray pr. sol. in HNO_3.

THE ACIDS.

Acids.

Lead Acetate.	CHARACTERISTIC REACTIONS.
Wh. pr. sol. in NaOH, in ammonium tartrate, in am. acetate.	Sulphates when heated with Na_2CO_3 in the reducing flame on charcoal give a hepar (Na_2S), which blackens silver, when moistened with water.
——	The potassium salt is difficultly soluble. Fluosilicates give with conc. H_2SO_4, HF and SiF_4.
White pr.	Sulphites reduce starch iodide, or $KMnO_4$, give a hepar with Na_2CO_3 on charcoal. Give SO_2 without separation of S, on warming with HCl.
White pr. on boiling becomes gray, $PbS+PbSO_4$.	Gives on warming with HCl, SO_2 and a pr. of S. Gives a hepar with Na_2CO_3 on charcoal. $Na_2S_2O_3$ dissolves Ag Cl.
White pr. sol. in NaOH, insol. in NH_4OH.	Added to a solution of ammonium molybdate in HNO_3, gives a yellow pr. of a ammonium phosphomolybdate; $MgSO_4+NH_4OH+NH_4Cl$ precipitate white cryst, $MgNH_4PO_4$
White pr. sol. in an excess of the pyrophosphate.	Does not coagulate albumen. Changed to H_3PO_4 by boiling with HNO_3.
White pr.	Coagulates albumen. Changed to H_3PO_4 by boiling with HNO_3.
White pr. sol. in HNO_3 insol. in $C_2H_4O_2$.	Yellow precip. with $(NH_4)_2MoO_4$. White precip. with $NH_4Cl+NH_4OH+MgSO_4$.
White pr. sol. in HNO_3 soluble in $C_2H_4O_2$.	Marsh's test gives an arsenic mirror. H_2S precipitates yellow As_2S_3 from the warm acid solution.
Yellow pr. sol. in NaOH.	Reduced to green Cr_2Cl_6 by warming with HCl and alcohol.
White pr. sol. in excess of the lead salt.	With glycerine and conc. H_2SO_4 on Pt wire, colors flame green. Turmeric paper dipped in the HCl solution becomes red on drying.
White pr. insol. in $C_2H_4O_2$.	Reducing agent. Very concentrated solutions evolve PH_3 on heating.
White pr. insol. in HNO_3.	Evolves CO_2 with Na_2CO_3 bead. Gives a skeleton of SiO_2 with $NaPO_3$ bead. SiO_2 separates on evaporating the acid solution to dryness.
White pr. sol. in HNO_3.	Effervesces with HCl giving CO_2 which renders lime water turbid.

I.—Inorganic

	BARIUM CHLORIDE.	CALCIUM CHLORIDE.	SILVER NITRATE.
Hydrofluoric Acid. HF.	White pr. sol. in HCl.	White, gelatinous pr., sol. in HCl, insol. in $C_2H_4O_2$.	——
Iodic Acid. HIO_3.	White pr. sol. in HCl.	——	White pr., sol. in NH_4OH. Reduced to AgI by SO_2.
Hydrochloric Acid. HCl.	——	——	White pr., insol. in HNO_3, soluble in NH_4OH, in KCN.
Hydrobromic Acid. HBr.	——	——	Yellowish white pr., difficultly sol. in NH_4OH.
Hydriodic Acid. HI.	——	——	Yellow precip. very difficultly sol. in NH_4OH.
Hydrocyanic Acid. HCN.	——	——	White precip. sol. in NH_4OH, in KCN.
Hydroferrocyanic Acid. H_4FeCy_6.	——	——	White pr. soluble in KCN, insoluble in HNO_3.
Hydroferricyanic Acid. H_3FeCy_6.	——	——	Orange pr. sol. in NH_4OH, in KCN, insol. in HNO_3.
Sulphocyanic Acid. HCNS.	——	——	White precipitate, difficultly sol. in NH_4OH.
Nitrous Acid. HNO_2.	——	——	White pr. sol. in hot water.
Hypochlorous Acid. HClO.	——	——	White pr. of Ag Cl.
Hydrosulphuric Acid. H_2S.	——	——	Black precipitate, soluble in hot HNO_3.
Nitric Acid. HNO_3.	——	——	——
Chloric Acid. $HClO_3$.	——	——	——

Acids.

LEAD ACETATE.	CHARACTERISTIC REACTIONS.
White precipitate, sol. in HNO_3.	With conc. H_2SO_4 gives HF, which etches glass.
White precipitate.	With KI and $C_2H_4O_2$, gives free I.
White precipitate, soluble in hot water.	With H_2SO_4 and MnO_2 gives Cl. With conc. H_2SO_4 and $K_2Cr_2O_7$ gives CrO_2Cl_2.
White precipitate, soluble in hot water.	Cl water liberates Br which dissolves in CS_2 to a reddish yellow solution.
Yellow precipitate, soluble in hot water.	Cl water, or nitrous acid, liberates I, which dissolves in CS_2 to a violet red solution, or colors starch paste blue.
White precipitate, soluble in HNO_3.	After warming with $FeSO_4$, Fe_2Cl_6 and NaOH, on adding HCl, Prussian blue remains undissolved. When warmed with yellow $(NH_4)S$, NH_4CNS is formed.
White precipitate.	Gives Prussian blue with ferric salts. Gives a red precipitate with Cu Salts.
No pr. in alkaline solutions, but on warming PbO_2 is precipitated.	Gives a dark blue precipitate with ferrous salts.
White precipitate.	Blood red color with Fe_2Cl_6, which vanishes on adding $HgCl_2$.
White pr. changing to orange, red and br'n on boiling.	Gives with $MnSO_4$ a brownish black pr. of MnO_2.
——	Gives a yellow color with meta-phenylene-diamine in acid solution.
Black precip, sol. in hot HNO_3.	Sulphides give a hepar with Na_2CO_3. See sulphates. Soluble sulphides give a violet color with Sodium nitroprussiate.
——	Mixed with $FeSO_4$ and poured on conc. H_2SO_4, gives a dark red zone. Colors a solution of brucin in H_2SO_4, red.
——	Chlorates deflagrate on charcoal. Added to conc. H_2SO_4 they give yellow ClO_2.

II.—Organic

	CALCIUM CHLORIDE.	SILVER NITRATE.
Formic Acid. CO_2H_2.	——	White precipitate in conc. solutions, becoming dark from reduction to Ag.
Acetic Acid. $C_2H_4O_2$.	——	White precipitate, soluble in hot water.
Oxalic Acid. $C_2O_4H_2$.	White precipitate, insoluble in $C_2H_4O_2$.	White precipitate, soluble in HNO_3.
Tartaric Acid. $C_4H_6O_6$.	White precipitate, soluble in NaOH, reprecipitated by boiling but dissolves again on cooling.	White precipitate with alkali salts, soluble in HNO_3 and in NH_4OH. Precipitate or mirror of Ag on warming.
Malic Acid. $C_4H_6O_5$.	White precipitate only in presence of strong alcohol.	White precipitate.
Citric Acid. $C_6H_8O_7$.	White precipitate, less soluble in hot than in cold water. Insoluble in NaOH, soluble in NH_4Cl.	White precipitate.

	FERRIC CHLORIDE.	SILVER NITRATE.
Benzoic Acid. $C_7H_6O_2$.	Flesh colored precipitate, soluble in HCl.	White precipitate, soluble in hot water.
Succinic Acid. $C_4H_6O_4$.	Pale red precipitate.	White precipitate.
Salicylic Acid. $C_7H_6O_3$.	Deep violet color.	White precipitate, soluble in hot water.
Gallic Acid. $C_7H_6O_5$.	Blue black precipitate.	Reduces to metallic Ag.
Tannic Acid. (Tannin.) $C_{14}H_{10}O_9$.	Blue black color.	White precipitate.

Acids.

LEAD ACETATE.	CHARACTERISTIC REACTIONS.
——	Reducing agent, with conc. H_2SO_4 gives CO.
——	Gives with Fe_2Cl_6 a reddish brown solution and a precipitate on boiling. Alcohol and conc. H_2SO_4 gives ethyl acetate, $C_2H_3O_2$ C_2H_5, recognized by its odor.
White pr. sol. in HNO_3 and in NH_4OH.	Heated with conc. H_2SO_4 gives CO_2 and CO. With dil H_2SO_4 and MnO_2 gives CO_2.
White pr. sol. in NaOH.	The acid K and NH_4 salts are difficultly soluble.
White pr. which melts under boiling water.	
White pr. sol. in ammonium citrate.	Tartaric, malic and citric acids prevent the precipitation of Fe_2Cl_6 by alkalies.

LEAD ACETATE.	CHARACTERISTIC REACTIONS.
White pr.	Melts at 121°.
White pr.	Melts at 180°.
White pr.	Melts at 156°.
——	Melts at about 200°. The alkaline sol. absorbs O.
Yellow pr.	Amorphous, with an astringent taste. It precipitates a solution of glue.

PART II.

SYSTEMATIC ANALYSIS.

Before beginning the analysis of an unknown substance the chemist should always consider the amount which he has at his disposal and how much he can use for each operation. Portions will be needed for the preliminary examination, for the detection of metals and for the detection of acids, and such amounts should be used for each of these operations that at least one-third will remain for cases of accident and for confirmatory tests.

Preliminary Examination.

The object of the preliminary examination is to obtain, if possible, some indication of the character of the substance under examination which shall enable the chemist to shorten the course of analysis. Thus, if a particular acid is indicated by the preliminary examination, the chemist can apply the test for that acid first and prove its presence or absence. Frequently much time may be saved in this way, but, in case the indications of the preliminary examination are not sustained by the special tests, it is usually advisable to apply the tests for the other acids in regular order.

The following tests are intended for the preliminary examination of solid substances. If the substance is a liquid, see page 59. If the substance is a metal or an alloy, only tests 1 and 3 need be applied, but special directions are given under 7 for the solution of such substances.

An outline of the preliminary tests is as follows:

1. Heat a little of the powdered substance in a small glass tube, closed at one end.
2. Put 2 cc. of con. H_2SO_4 in a test tube, add a little of the dry substance, and, if there is no action, heat cautiously.
3. Heat a small portion on charcoal in the R. F.
4. Heat a small portion on charcoal with Na_2CO_3 in the R. F.
5. Test with the borax bead in the O. F.
6. Moisten a little with con. HCl and test in the flame with a clean platinum wire.
7. Treat the substance with solvents in the following order: water, dil. HCl, conc. HCl, dil. HNO_3, aqua regia.

The results to be looked for, and the indications of various results are as follows:

1. *Heating in a closed tube.*

	Result.	Indication.
a.	Carbonization—recognized by blackening and the disengagement of water and gases with a "burnt odor"	Organic matter.
b.	Changes color.	
	Yellow when hot, white when cold	ZnO.
	Yellowish brown when hot, light yellow when cold	SnO.
	Yellowish red when hot, yellow when cold	PbO.
	Orange yellow when hot, ale yellow when cold	Bi_2O_3.
	Brownish red to black when hot, brownish red when cold	Fe_2O_3.
	Yellow to dark orange and fuses when very hot	$PbCrO_4$.
c.	Gives water. It may react alkaline from NH_4 salts or acid from a volatile acid	Water of crystallization, or mechanically held, or from the decomposition of a hydroxide.
d.	Yields a gas.	
	O—Enflames a glowing splinter	Clorates, peroxides and nitrates.
	SO_2—Known by the odor	Sulphates, etc.
	NO_2—Brownish red color and peculiar odor	Nitrates.
	CO_2—Renders turbid a drop of lime water	Carbonates, oxalates and organic matter.
	CO—Burns with a blue flame	Oxalates, formates.
	Cl, Br, I—Known by odor and color	Chlorides, bromides or iodides.
	C_2N_2—Burns with a crimson flame	Cyanides.
	H_2S—Blackens lead acetate paper	Sulphides with water.
	NH_3—Turns red litmus blue	NH_4 salts, cyanides.
e.	Gives a sublimate.	
	Black	As.
	Mirror or small globules	Hg.
	Black, becomes red when rubbed	HgS.
	Black, violet vapors	I.
	Reddish brown, yellow on cooling	S and persulphides
	White sublimate	NH_4 salts, As_2O_3, HgCl, $HgCl_2$, Sb_2O_3, some organic acids.
	Yellow	As_2S_3.
	Yellow, becomes red when rubbed	HgI_2.

2. *Heated with con.* H_2SO_4.

RESULT.	INDICATION.
CO_2—Renders turbid a drop of lime water	Carbonates, oxalates.
CO—Burns with a blue flame—usually accompanied by an odor of hydrocyanic acid	Cyanides, ferro- and ferri-cyanides.
HF—Etches the test tube, producing SiF_4 which gives a precipitate of silicic acid with a drop of water	Fluorides.
H_2S—Known by odor	Sulphides.
SO_2—Known by odor	Sulphites.
HCl—Gives a white precipitate with $AgNO_3$	Chlorides.
Br, I—Known by odor and color	Bromides, iodides.
Cl, ClO_2—Known by yellow color and bleaching litmus. ClO_2 is explosive	Hypochlorites, chlorates.
NO_2—Brownish red color and peculiar odor	Nitrates, nitrites.
$C_2H_4O_2$—Odor of vinegar	Acetates.

3. *Heated on charcoal.*

RESULT.	INDICATION.
a. Melts and runs into the charcoal	Alkali salts.
b. Leaves an infusible residue which reacts alkaline	Ba, Sr, Ca, Mg.
c. Leaves a residue which when moistened with $Co(NO_3)_2$ and heated again is: Blue,	Al [SiO_2 slight].
Green,	Zn.
Rose color	Mg.
d. Deflagrates with the charcoal	Chlorates, nitrates
e. Forms an incrustation.	
White, with odor of garlic	As.
White, nearer the test	Sb.
Yellow when hot, white when cold	Zn.
Light yellow when hot, white when cold, close to the test and not volatile	Sn.
Lemon yellow when hot, sulphur yellow when cold	Pb.
Orange yellow when hot, lemon yellow when cold	Bi.
Reddish brown to orange	Cd.
Dark red	Ag.

4. *Heated on charcoal with* Na_2CO_3.

RESULT.	INDICATION.
a. Yields metallic globules without incrustation	Au, Cu.
b. Yields metallic globules with incrustation.	
Malleable beads	Ag, Pb, Sn.
Brittle beads	Sb, Bi.
c. Yields magnetic particles	Fe, Co, Ni.
d. In the O. F. gives	
a yellow mass	Cr.
a green mass	Mn.

5. *Borax bead in O. F.*

Result.		Indication.
Hot.	Cold.	
Blue	Blue	Co.
Green	Blue—in R. F. red	Cu.
Green	Green	Cr.
Brownish red . .	Light yellow—in R. F. Green	Fe.
Violet	Reddish brown—in R. F. grey	Ni.
Yellowish brown	Light yellow	Bi.
Violet red . . .	Amethyst red–in R.F. colorless	Mn.

6. *Flame test.*

Result.	Indication.
Yellow	Na.
Violet	K.
Carmine red	Li.
Bright red	Sr.
Brick red	Ca.
Green	Ba, boric acid.
Blue to green	Cu.

7. *Solution.*

A. The substance is neither a metal nor an alloy.

a. Put ¼ of a gram of the substance into a test tube, add 5 to 10 cc. of water and boil. If the substance dissolves giving a solution with a neutral or acid reaction, proceed at once to the detection of the metals, beginning with Group I. See also p. 60. If the reaction of the solution is alkaline, see p. 60.

If the substance proves to be wholly or partly insoluble in water, filter, leaving the residue in the test tube, if possible, and evaporate a few drops of the filtrate in a porcelain dish. If a residue remains, the substance is at least partly soluble in water and should be washed in hot water as long as anything dissolves. Treat the aqueous solution as directed above, and the insoluble residue by b.

b. To the residue insoluble in water add three or four cc. of dil. HCl and boil. If this fails to dissolve it, decant the liquid into another test tube and boil the residue with three or four cc. conc. HCl. If it dissolves, add the solution to that in the other test tube and treat the whole for metals, beginning with Group II, noticing especially what is said about free acid.

During the treatment with HCl, CO_2 or H_2S may be evolved, indicating a carbonate or a sulphide, Cl, indicating a dioxide or a chromate, or HCN indicating a cyanide.

Sometimes there will remain, on treatment with acid, a residue of sulphur, known by its color, light specific gravity, and by drying and burning it, or of silicic acid, which can be known by the tests with the Na_2CO_3 and $NaPO_3$ beads. (See page 66.) Silicic acid must be rendered insoluble by evaporating to dryness, avoiding heating the residue much above 100°. Then moisten with conc. HCl, dissolve in hot water, and filter.

c. If the substance is insoluble in HCl, try to dissolve it in HNO_3, boiling as before. If NO or NO_2 is evolved, this indicates that an oxidation is taking place. If the substance dissolves, test the solution for metals, beginning with Group I, but it should be remembered that it is much more difficult to precipitate the metals of Group II from a solution in HNO_3 than from a solution in HCl.

d. If the substance is insoluble in HCl and in HNO_3, try to dissolve it in boiling aqua regia. If it dissolves, test the solution for metals, beginning with Group II. If the substance is insoluble in aqua regia, treat as directed on p. 73.

B. Metals or Alloys.

Treat the substance with HNO_3 of sp. gr. 1.20 and boil, if necessary.

a. The metal or alloy dissolves completely. Au, Pt, Sn and Sb are absent.

b. A metallic residue is left. Dissolve it in aqua regia and test for Au and Pt.

c. A white residue is left. This may consist of metastannic acid, H_2SnO_3, or metantimonic acid, $HSbO_3$, or of certain nitrates which are with difficulty soluble. Add water and warm to dissolve the nitrates and filter. Test the filtrate for metals, beginning with Group I. Wash the residue thoroughly and test a portion of it for antimony with Zn, Pt, and dil. HCl as in **(15)**. Test the other portion for tin by boiling it with conc. HCl and an iron nail, diluting, filtering and adding $HgCl_2$.

d. Sometimes iron and other metals leave a residue of carbon or graphite.

Preliminary Examination of Solutions.

Evaporate a small portion in a porcelain dish and, if a residue remains, submit it to the preliminary examination as directed above.

Test the solution with litmus paper, blue and red.

a. An acid reaction may be caused by a free acid or by a salt of a heavy metal. In the latter case a permanent precipitate will be formed if a little of the solution is poured into a watch glass and stirred with a glass rod which has been moistened with a dilute solution of Na_2CO_3.

b. An alkaline reaction is usually caused by a hydroxide, sulphide or carbonate of an alkali or alkali earth metal. See next page.

Smell of the fluid to determine whether alcohol, ether, &c., are present.

DETECTION OF METALS.

The systematic course to be followed for the detection of metals has already been given, and only a few words are necessary here.

In the examination of a solution it is advisable to add the group reagents to separate *small portions* at first, so as to avoid the addition of the reagents to the whole solution, in case the group looked for is absent.

At first HCl should be added to the whole solution to distinctly acid reaction. Then, if no precipitate is produced, a *small portion* of the solution may be warmed and tested with H_2S. If Group II is absent, a *second small portion* may be tested for Group III by adding NH_4Cl, NH_4OH and $(NH_4)_2S$. If Group III is absent, $(NH_4)_2CO_3$ may be added to the same portion, but if this produces no precipitate, it is best to test a new portion for traces of Ca by adding NH_4OH and $(NH_4)_2C_2O_4$, and another portion for Ba by adding NH_4OH and a drop of dil. H_2SO_4 and warming. If both are absent, the same portion may be tested for Mg with Na_2HPO_4. If Mg is absent, a new portion must be tested for Na, K and NH_4.

It is possible in the manner indicated to determine very quickly to what group a substance belongs. The order and method of applying the group reagents should be learned by every chemist so that he can use them without reference to the text book.

ALKALINE SOLUTIONS.

If the original solution has an alkaline reaction, HCl may precipitate AgCl, $PbCl_2$, $PbSO_4$, As_2S_3, Sb_2S_3, SnS_2, HgS, CuS, NiS, H_2SiO_3, or S.

For full directions in such cases reference must be had to larger works on qualitative analysis. Sulphur is recognized by its white or yellow color and by the fact that it passes readily through the filter so that solutions from which it has been precipitated cannot be filtered clear until after protracted boiling or evaporation nearly to dryness. Sulphur indicates the presence of a polysulphide or of a thiosulphate, ("hyposulphite").

The sulphides soluble in alkaline solution are colored, and those most likely to be met with are yellow. Ag and Pb cannot be present in the same solution with such sulphides.

$PbSO_4$ and $PbCl_2$ may be dissolved in ammonium acetate and the solution tested for Pb with H_2S, for SO_4 with $BaCl_2$ and HCl, and for Cl with HNO_3 and $AgNO_3$.

After the removal of lead, AgCl and AgCN can be dissolved in KCN and the Ag can be precipitated from the solution by $(NH_4)_2S$. The Ag_2S may be dissolved in hot HNO_3 and the Ag reprecipitated with HCl.

Silicic acid usually separates in the gelatinous form. It may be tested and rendered insoluble, as directed on p. 66.

GROUPS OF THE ACIDS.

The acids may be divided into four groups. The first group is further divided into four divisions.

Group I.—Acids which are precipitated from neutral solutions by barium chloride.

a. Acids containing metals which will be detected in the examination for metals.
Chromic acid, arsenious acid, arsenic acid.

b. Acids precipitated from acid solutions by barium chloride. *Sulphuric acid, [hydrofluosilicic acid.]*

c. Acids whose barium salts are soluble in HCl without apparent decomposition.
Phosphoric acid, boric acid, oxalic acid, hydrofluoric acid.

d. Acids whose barium salts are decomposed by HCl.
Carbonic acid, sulphurous acid, thiosulphuric acid, silicic acid.

Group II.—Acids which are precipitated from solution in dilute acids by silver nitrate.

Hydrochloric acid, hydrobromic acid, hydriodic acid, hydrocyanic acid, hydroferrocyanic acid, hydroferricyanic acid, hydrosulphuric acid (H_2S), [*hypochlorous acid, chlorous acid, nitrous acid, sulphocyanic acid.*]

Group III.—Acids whose barium and silver salts are soluble in water.

Nitric acid, chloric acid, acetic acid, [*perchloric acid, permanganic acid.*]

Group IV.—Organic acids which carbonize on heating. *Tartaric acid, citric acid, acetic acid.** [*Malic acid, succinic acid, benzoic acid, lactic acid, propionic acid, butyric acid, and hundreds of others.*]

ANALYSIS FOR ACIDS.

It is usually best to complete the examination for metals before beginning that for acids. Then, on examination of the table of solubilities on page 90, the chemist will frequently find that many of the tests for the acids can be omitted. Thus, if barium has been found in a substance soluble in water, all of the acids of Group I must be absent.

The preliminary examination, especially the heating in a closed tube, frequently gives a clue to the acid present. In such cases the test for the acid suspected may be applied first. In case no clue has been obtained, it will usually be best to follow the order given below.

The tests for the acids are surest to give satisfactory results when applied to solutions containing only alkali metals. Such a solution should be prepared as follows, if metals of the first four groups are present: Dissolve† the substance in water or in the least possible amount of cold dil. HNO_3. Add a strong solution of Na_2CO_3 until the reaction is strongly alkaline, and boil in a porcelain dish. If ammonium salts are present the solution must be concentrated to one-half or one-third of the original volume, adding more Na_2CO_3, if necessary, to keep it alkaline. This is for the purpose of expelling NH_4 which would prevent the precipitation of Ag, Cu, &c. Filter and use the filtrate for the following tests. It may still contain As and Sb and, in some cases, traces of other metals, but the cases in which these are likely to interfere are mentioned below under the special tests.

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*Acetates sometimes give acetone with very little blackening.

†If the substance is insoluble in cold dil. HNO_3 but is soluble in concentrated acids or in aqua regia, it is advisable to apply tests for sulphuric, hydrofluoric, silicic, hydriodic and hydrosulphuric acids to the original substance. If the acid is not detected in this way, fuse a portion of the substance with Na_2CO_3 on Pt foil, or, if any metal of the first two groups except mercury is present, on charcoal. Boil the fused mass with water, filter and test the filtrate for acids as directed.

GROUP I.

The acids of division *a* will have been detected in the examination for metals. If the original substance dissolves in water giving a neutral or alkaline solution and $BaCl_2$ produces no precipitate in the concentrated solution, the acids of GROUP I are absent. Pass to GROUP II. If a sodium carbonate solution has been prepared, apply to it the test for sulphuric acid. If that is absent, boil the solution to which $BaCl_2$ and HCl have been added, to expel CO_2, and add NH_4OH. A precipitate must be caused by some acid of GROUP I but the absence of a precipitate cannot be taken as a proof of the absence of boric acid or of oxalic acid.

Tests for hydrofluoric acid, for the acids of division *d* and also for hydrosulphuric acid must be applied to the original substance.

Sulphuric acid.—Add to a small portion of the solution dil. HCl to acid reaction (if this precipitates AgCl, filter) and add $BaCl_2$. A white pulverulent precipitate is $BaSO_4$. (See, however, the statements under hydrofluoric and sulphurous acids.)

All sulphates when heated with Na_2CO_3 on charcoal in the R. F. form a "hepar" containing Na_2S. If the fused mass is placed on a silver coin and moistened with water, a black stain is produced.

Free H_2SO_4 may be detected in a solution by dipping a piece of filter paper in it and drying it on the water bath, when the paper will be charred by the concentrated acid, if present.

Phosphoric acid.—Add to another portion of the solution dil. HCl to acid reaction, (if As or Sb are present, warm and precipitate with H_2S) boil to expel CO_2, then add NH_4Cl, NH_4OH and $MgSO_4$. A white crystalline precipitate is $MgNH_4PO_4$. If this is obtained, confirm as follows: Dissolve a little of the original substance in dil. HNO_3 (remove As, if necessary) and add a drop of the solution to 1 cc. of $(NH_4)_2MoO_4$ dissolved in nitric acid ("molybdic solution"). If a yellow precipitate does not form after waiting a short time and warming very gently, add more of the solution to be tested but never more than a volume equal to that of the molybdic solution taken. The yellow precipitate is ammonio-phospho molybdate, $(NH_4)_3PO_4 + 10\ MoO_3 + 1\frac{1}{2}\ H_2O$. The reason why the solution to be tested must be added so carefully is because the precipitate is soluble in phosphoric acid. Arsenic acid gives similar precipitates both with magnesia mixture and with molybdic solution.

Boric acid.—Add to a small portion of the solution, HCl just to acid reaction. Dip a piece of turmeric paper in the solution and dry it on the water-bath. If boric acid is present, the turmeric paper will turn red. Care must be taken not to mistake the brown color caused by an alkaline solution, or the blackish brown color produced if too much hydrochloric acid is present, for the red color characteristic of boric acid.

If chloric acid is present, the Na_2CO_3 solution must be evaporated and the residue ignited to faint redness to destroy it. The residue may then be dissolved in water and tested with HCl and turmeric as directed.

If a borate is mixed with a little conc. H_2SO_4 and alcohol is added, on setting fire to the latter it will burn with a green flame. Copper, if present, must first be removed by H_2S.

Oxalic acid.—If metals of the first three groups are present in the substance, these will frequently pass into the sodium carbonate solution as double oxalates of sodium with these metals. Such metals must be removed from the solution by H_2S before the test for oxalic acid is applied. Then acidify with acetic acid and boil till CO_2 and H_2S (if used) are expelled. Add $CaSO_4$ A white pulverulent precipitate is CaC_2O_4. Hydrofluoric acid, if present, also gives a precipitate, but CaF_2 is gelatinous and so transparent that it can hardly be seen.

If an oxalate is boiled with MnO_2 and dil. H_2SO_4, it is oxidized to CO_2 which causes effervescence and may be identified by passing it through lime water. Carbonates, if present, may be decomposed by boiling with dil. H_2SO_4 for some time before adding the MnO_2.

Oxalates of Na, K, Ca, Sr and Ba are converted into carbonates by gentle ignition.

Hydrofluoric acid.—The tests for hydrofluoric acid must be applied to the original solid substance. Mix a small portion of it with $KHSO_4$. Dip in the mixture a borax bead prepared in the usual way, and test in the Bunsen flame. A green flame, appearing only for an instant, indicates the formation of BF_3. Always confirm by mixing some of the substance with enough conc. H_2SO_4 in a lead dish to form a thin paste, laying over the dish a piece of glass covered with beeswax in which letters have been traced with a pointed piece of wood, and heating very gently, but not hot enough to melt the wax. If the substance is a fluoride, lines will be etched on the glass.

If a fluoride is dissolved by means of HCl in a glass vessel, H_2SiF_6 is formed, which gives with $BaCl_2$ a precipitate insoluble in HCl and sometimes mistaken for $BaSO_4$.

For the detection of fluorine in silicates, reference must be had to Fresenius' Qualitative Analysis.

Carbonic acid.—Most carbonates are converted into chlorides by dil. HCl. The conversion is accompanied by effervescence caused by the escape of CO_2. A very few carbonates require a concentrated acid or warming for their decomposition. The CO_2 may be identified as follows. Fit a test tube with a cork through which is inserted a bent tube fitting tightly. Put a little of the substance to be tested into the test tube and add 2 or 3 cc. of dil. HCl. Insert the cork, boil, and conduct the steam and gases through 2 or 3 cc. of clear lime water in another test tube. If a precipitate is formed, it must be $CaCO_3$.

Sulphurous and **Thiosulphuric** ("hyposulphurous") **acids.**

Both sulphites and thiosulphates evolve SO_2 on warming with dil. HCl. Sulphur dioxide is readily recognized by its odor. It is also a reducing agent and acid solutions containing it instantly decolorize a solution of potassium permanganate.

Sulphites are easily oxidized to sulphates and a solution of a sulphite will usually give the test for sulphuric acid.

Thiosulphates give a white precipitate of S at the same time that SO_2 is evolved.

Silicic acid.—The only common silicates soluble in water are those of K and Na. If HCl is added drop by drop to a strong solution of a silicate, most of the silicic acid separates in the gelatinous form. If the acid solution is evaporated completely to dryness, but with care not to heat the residue after it is dry, on moistening it with conc. HCl (or HNO_3 if Ag is present) and then treating it with hot water the silicic acid will remain undissolved. The same method serves for the detection and separation of silicic acid in silicates which are decomposed by conc. HCl or conc. HNO_3. The silica obtained as above, or the silica in any silicate will dissolve with effervescence in a bead of Na_2CO_3, but both are insoluble in a bead of sodium metaphosphate (obtained from $NaNH_4HPO_4$, "microcosmic salt.")

For the analysis of silicates see page 74.

GROUP II.

Add to another portion of the Na_2CO_3 solution, or of the solution of the substance in water, dil. HNO_3 to acid reaction, and $AgNO_3$. If no precipitate forms, the acids of Group II are absent. Pass at once to nitric acid. If a precipitate forms, the color may give an indication of its character AgCl, AgCN, $AgNO_2$ and $Ag_4FeC_6N_6$ are white. AgBr is yellowish white. AgI is yellow. $Ag_3FeC_6N_6$ is brownish red. Ag_2S is black.* Test for these acids as follows:

Hydrobromic and Hydriodic acids.—Add to a small portion of the solution dil. H_2SO_4 to strongly acid reaction, then add a drop of carbon disulphide, which will sink to the bottom, and a single drop of chlorine water, and shake. The Cl will combine with the hydrogen of the HI or HBr and set iodine or bromine free. The bromine will color the CS_2 yellow or reddish yellow while the iodine colors it violet red. If iodine is found, add Cl water carefully, shaking after each addition. The Cl will combine with the iodine to form colorless iodine chloride, ICl_3. When the color caused by the iodine has disappeared the CS_2 will be colorless if bromine is absent but yellow if it is present. The tests for Br and I are best applied to dilute solutions.

If the original substance was not soluble in cold dil. HNO_3, a fresh portion should be tested for iodine by putting it in a test tube with a drop of CS_2 and a little dil. H_2SO_4. Then add a drop of Cl water and shake. If the substance contains an iodide the characteristic color will be seen in the CS_2.

A further test for iodine may be made by adding a solution of starch, dil. H_2SO_4 and a drop of Cl water to the solution.

If very minute quantities of iodine are to be detected,

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*The colors of the following precipitates formed in neutral or alkaline solutions will sometimes be found useful. $Ag_2C_2O_4$, Ag_2SiO_3 and $Ag_2B_4O_7$ are white. Ag_3PO_4 and Ag_3AsO_3 are yellow. Ag_3AsO_4 is brownish red. Ag_2CrO_4 is purple red.

KNO_2 should be added to the acid solution instead of chlorine water, since an excess of nitrous acid does not destroy the color caused by the iodine.

Hydrocyanic Acid.—Dissolve a little of the original substance in water or in dil. HCl without warming. Add a few drops of $FeSO_4$ and of Fe_2Cl_6. These should produce no precipitate if the solution is acid. (If a blue precipitate is formed, $H_4FeC_6N_6$ or $H_3FeC_6N_6$ is present. In that case see Fresenius.) Add NaOH to alkaline reaction and warm, then acidify with dil. HCl. If hydrocyanic acid was originally present it will be converted, by the treatment directed, into hydroferrocyanic acid and on acidifying with HCl a precipitate of Prussian blue $Fe_4Fe_3C_{18}N_{18}$ will be obtained.

Hydrocyanic acid can frequently be detected by its odor in neutral or acid solutions. Great care must always be exercised in working with it as it is volatile and is a deadly poison.

Hydroferrocyanic Acid. Add to the acid solution a drop of Fe_2Cl_6. A dark blue precipitate is Prussian blue $Fe_4(FeC_6N_6)_3$.

Hydroferricyanic Acid.—Add to the acid solution a drop of $FeSO_4$ (freshly prepared by dissolving an iron nail in dil. H_2SO_4.) A deep blue precipitate is Turnbull's blue, $Fe_3(FeC_6N_6)_2$. Under the same circumstances, hydroferrocyanic acid gives a *light blue* precipitate.

If the precipitate with $AgNO_3$ was white, it is, of course, unnecessary to test for hydroferricyanic acid.

Hydrochloric Acid. If the precipitate with silver nitrate in acid solution is white and HI, HBr, HCN, HNO_2 and $H_4FeC_6N_6$ are absent, it must consist of AgCl.

Hydrochloric acid may be detected in the presence of HI and HBr as follows: Add to the acid solution a little $AgNO_3$ but not enough to precipitate all of the acids present. Filter and test a small portion of the filtrate for Br and I with CS_2 and chlorine water. If they are found to be absent, test the remainder of the filtrate for HCl with $AgNO_3$. The test depends on the fact that if $AgNO_3$ is added to a solution containing HCl, HBr and HI, the HBr and HI may be entirely precipitated, while a portion of the HCl remains in solution.

If HCN is present, and it is desired to test for the other acids of the group, add $AgNO_3$ in sufficient amount to precipitate all the acids present, filter, wash, dry the precipitate and ignite it in a porcelain crucible to faint redness. The AgCN will be decomposed. When cold add to the contents of the crucible a little dil. H_2SO_4 and a fragment of zinc. The nascent hydrogen will reduce the AgCl, etc., to metallic Ag while HCl, HBr and HI will pass into solution. Filter and test the filtrate for these acids.

Nitrous Acid.—Nitrites in concentrated solutions evolve reddish brown fumes of N_2O_3 and NO_2 when acidified with dil. H_2SO_4. If to a very dilute solution of a nitrite, a few drops of dil. HCl and a few drops of a solution of meta-phenylene-diamine chloride, $C_6H_4(NH_2HCl)_2$, be added, a yellow color develops at once or after some time, owing to the formation of triamido-azobenzene, $C_{12}H_{13}N_5$. This reaction is especially suited for water analysis.

Solutions of naphthylamine chloride and sulphanilic acid when both added to an acid solution of a nitrite give a rose color which can be more clearly seen than the preceding test in the case of colored or turbid waters.

Nitrous acid gives the same reactions as nitric acid with brucine and sulphuric acid, or with ferrous sulphate.

Hypochlorous Acid.—Hypochlorites evolve chlorine, which bleaches litmus paper, when warmed with dil. H_2SO_4. With $MnSO_4$ hypochlorites give a brownish black precipitate of MnO_2.

Hydrosulphuric Acid. Sulphides which are soluble in water or in HCl, evolve hydrosulphuric acid (H_2S) on treatment with that acid. Hydrogen sulphide can be recognized by its odor and by its blackening a paper moistened with $Pb(C_2H_3O_2)_2$.

All sulphides when fused on charcoal with Na_2CO_3 yield Na_2S. If the fused mass is laid on a silver coin and moistened with water, a brown or black stain of Ag_2S will be produced. Other compounds containing sulphur (sulphates, etc.,) give the same reaction and care must be exercised to distinguish between them.

By solution in aqua regia the sulphur of many sulphides is converted into sulphuric acid, which may then be detected in the usual way. This method can be used to detect sulphur in cast iron and in other similar cases.

GROUP III.

Nitric Acid.—If the Na_2CO_3 solution was prepared by first dissolving the substance in HNO_3, a fresh portion of the original substance must be boiled with Na_2CO_3 (in case metals of the first four groups are present) and the filtered solution must be tested for HNO_3. Add to the solution dil. H_2SO_4 to acid reaction and a little $FeSO_4$. Then pour conc. H_2SO_4 carefully down the side of the tube so that it will run to the bottom and leave the solution to be tested floating on top. If nitric acid is present, the $FeSO_4$ will reduce it to nitric oxide which will dissolve in the excess of $FeSO_4$ forming a reddish brown ring at the juncture of the two fluids. On shaking gently the ring increases, but on mixing the fluids the color usually disappears. Nitrous acid shows the same reaction.

Chlorates interfere with the reaction. If present, the alkaline solution must be evaporated to dryness and the residue gently ignited to destroy them. Iodides also interfere and may be removed from the solution by means of silver sulphate.

Chloric Acid.—Pour 1 or 2 cc. of conc. H_2SO_4 into a dry watch glass and put into it a small fragment of the original dry substance. A yellow color and the peculiar odor of chlorine peroxide, ClO_2, proves the presence of a chlorate. A reddish yellow color with a different odor will frequently be observed, if both a nitrate and a chloride are present. In cases of doubt, compare the action of a mixture of the substance with $KClO_3$ with the action of the substance alone.

All chlorates when heated give oxygen and leave a chloride. All chlorates deflagrate when heated on charcoal.

Acetic Acid.—Put a little of the dry substance, or a concentrated solution, into a test tube with 2 or 3 cc. of alcohol and an equal volume of conc. H_2SO_4 and warm gently. If an acetate is present, ethyl acetate C_2H_5, $C_2H_3O_2$, will be formed and may be recognized by its fragrant and characteristic, "fruity" odor. It need hardly be said that this test should not be applied in the presence of a chlorate.

GROUP IV.

The presence of organic acids will have been detected, if there are any in the substance, by the carbonization on heating in a closed tube. If the organic acid is non-volatile (e. g. tartaric, citric or malic acid), boiling with Na_2CO_3 will fail to precipitate completely metals of the first three groups and they must be separated in another way before the tests for the acids of the group are applied. Dissolve some of the substance in water, or, if necessary, with the least possible amount of HCl, keeping the solution as concentrated as possible.

If the metal belongs to the first or second group, precipitate it with H_2S, if to the third group, by adding NH_4OH and passing H_2S or adding $(NH_4)_2S$. (Al and Cr cannot be removed in this way.) After filtering, make the solution slightly acid with HCl if it is not already so, and boil in a porcelain dish to expel H_2S.

Tartaric Acid.—Add NH_4OH just to alkaline reaction, then NH_4Cl and $CaCl_2$, shake vigorously and allow to stand for ten minutes. If a precipitate forms, filter and test the filtrate for citric acid. Digest the precipitate with a cold solution of NaOH. If calcium tartrate is present, it will dissolve. Dilute slightly, filter and boil the filtrate. If a precipitate forms, tartaric acid is indicated. Filter hot, wash, introduce the precipitate into a test tube, add *one* drop of NH_4OH and a little $AgNO_3$ and warm. If tartaric acid is present, it will be oxidized by the $AgNO_3$ and the latter will be reduced to metallic silver which will give a black precipitate, or form a silver mirror on the walls of the tube.

Tartaric acid and tartrates, when heated, give a characteristic odor resembling that of burnt sugar. The odor is different from that given by citric acid.

Acid ammonium tartrate and acid potassium tartrate (cream of tartar) are difficultly soluble in cold water and are precipitated on adding HCl to the neutral ammonium or potassium salt, or on adding NH_4Cl or KCl to a slightly acid solution of a tartrate. The precipitation is aided by vigorous shaking.

Citric Acid.—To the solution in which $CaCl_2$ has produced no precipitate or which has been filtered from the precipitate, add 3 volumes of alcohol. If a precipitate forms, filter, wash with alcohol, dissolve on the filter with a little dil. HCl, add NH_4OH to the filtrate to alkaline reaction and boil for some time. A precipitate is calcium citrate, $Ca_3(C_6H_5O_7)_2$.

INSOLUBLE SUBSTANCES.

The most common of these substances are: $PbSO_4$; $AgCl$, $AgBr$, AgI, $AgCN$; SnO_2; Al_2O_3, Cr_2O_3, $FeCr_2O_4$; $BaSO_4$, $SrSO_4$ ($CaSO_4$), CaF_2; SiO_2 and many silicates; $Ag_4FeC_6N_6$, $Fe_4Fe_3C_{18}N_{18}$ and some other ferrocyanides and ferricyanides; S and C.

Examine carefully the physical properties and endeavor to obtain a clue to the composition of the substance in that way. If the preliminary examination already given has not furnished any clear indication of the character of the substances, apply the following tests.

a. Mix a little of the finely powdered substance with Na_2CO_3 and a drop of water and heat on charcoal in the reducing flame. Compounds of Pb, Ag, and Sn will give metallic globules. Sometimes these can be more easily found by cutting out the fused mass with a little of the charcoal adhering to it, powdering in a mortar and carefully washing the charcoal away with water, when the heavy metallic particles will remain. In the case of SnO_2, the reduction can be aided by adding a little dry KCN to the mixture before heating.

Warm the metallic globules or particles with 2 or 3 drops of dil. HNO_3. Ag and Pb will dissolve, while Sn will give white insoluble H_2SnO_3. If the bead dissolves, add a drop of dil. HCl. A precipitate insoluble in NH_4OH is $PbCl_2$ but if soluble in NH_4OH it is AgCl. According to the result obtained, treat as follows:

$PbSO_4$ and $PbCl_2$. If lead is found, dissolve the substance in ammonium acetate. Precipitate the lead from a part of the solution with H_2S. Test another portion of the solution for Cl with $AgNO_3$ and HNO_3 and another for H_2SO_4 by adding $BaCl_2$ and HCl.

Silver Salts.—If silver is found, put the solid subsubstance into a porcelain crucible or dish, add a fragment of zinc and dil H_2SO_4. After the action has continued for some time, pour off the solution and test it for chlorine, bromine, and iodine in the usual way. Wash the residue of metallic silver in the dish, dissolve it in HNO_3 and prove the presence of Ag as usual.

Stannic Oxide.—If tin has been found, powder the substance very finely and fuse it in a porcelain crucible with six times its weight of a mixture of equal parts of Na_2CO_3 and S. On treating the fused mass with water, the metals of Sub-Group A of Group II, and the metals of Group III will remain undissolved, as sulphides, while the Sn will dissolve as a sulpho salt and, after filtering, may be precipitated as SnS_2 by adding dil. HCl.

b. **Silicates.**—Prepare a bead of Na_2CO_3 and, holding a little of the finely powdered substance near the flame, dip the hot bead immediately into it. If the substance dissolves with effervescence, SiO_2 or a silicate is indicated. Test the substance also with a bead of $NaPO_3$ (from $NaNH_4HPO_4$). If a silicate, silica will float in the bead, undissolved.

If the substance is a silicate, powder it very finely and fuse it on platinum foil with four parts of a mixture of K_2CO_3 and Na_2CO_3. Put the fused mass in a porcelain dish, add dil. HCl in sufficient quantity to entirely decompose the carbonates, digest at a gentle heat till the mass is entirely disintegrated and then evaporate to perfect dryness, taking care, however, that the dry residue is not heated much above 100°. When cold, moisten the residue with conc. HCl, then dissolve in hot water, filter from the separated silica and test the filtrate for metals in the usual way.

In order to detect K and Na in silicates, mix some of the finely powdered substance with an equal weight of NH_4Cl and six times its weight of $CaCO_3$ and heat to redness on platinum foil for ten minutes. The mass should sinter together but not fuse. Boil the mass with water, filter, precipitate the Ca from the filtrate with

NH_4OH and $(NH_4)_2CO_3$, filter, evaporate to dryness, ignite till ammonium salts are completely expelled and test for Na and K in the usual way.

c. **Salts of Ca, Sr, and Ba.**—Moisten a little of the substance with conc. HCl and test in the flame with a clean platinum wire. Ba, Sr, and Ca can usually be detected in this way. Test also for S by fusing a little of the substance with Na_2CO_3 on charcoal in the reducing flame. Lay the fused mass on a silver coin, and moisten with water. If sulphates are present, they will be reduced to sulphides, which will produce a black or brown stain on the silver.

$BaSO_4$ and $SrSO_4$ are fused on platinum foil with K_2CO_3 and Na_2CO_3. On boiling the fused mass with water, $BaCO_3$ or $SrCO_3$ will remain undissolved. Filter and wash *thoroughly*. Then dissolve in $C_2H_4O_2$ and analyze as usual. The sulphuric acid can be detected in the filtrate from the carbonates by adding HCl and $BaCl_2$.

$CaSO_4$ is sufficiently soluble in water so that it may be detected by the regular course of analysis.

CaF_2, when finely powdered and boiled for some time with dil. HCl, dissolves in sufficient amount so that, on filtering and analyzing the solution for metals as usual, Ca will be found in the precipitate for Group III.

d. **$FeCr_2O_4$, Cr_2O_3 and Al_2O_3.**—If no metal of the first two groups is present, fuse a little of the finely powdered substance on platinum foil with Na_2CO_3 and $KClO_3$. If Cr is present, a yellow solution containing K_2CrO_4 will be obtained on boiling the fused mass with water. The solution, after filtering if necessary, may be acidified with HCl, evaporated to dryness to separate silica, the residue moistened with conc. HCl, dissolved in hot water and filtered. The filtrate may be analyzed as usual. A residue remaining on boiling the fused mass with water may be dissolved in HCl and the solution analyzed.

Cr_2O_3 and Al_2O_3 may be treated in essentially the same way but in the case of Al_2O_3 the solutions will be colorless.

e. **Ferrocyanides and Ferricyanides** are decomposed by boiling with NaOH, into the hydroxide of the metal and $Na_4FeC_6N_6$ or $Na_3FeC_6N_6$. The latter are soluble in water and can be washed out. The hydroxide can then be dissolved in HCl or HNO_3.

f. **Sulphur and carbon** can usually be recognized by their familiar properties and by burning them.

APPENDIX.

Form of Record.

It is essential that in performing the preliminary experiments such a record shall be kept of the work that afterwards it will be possible, by referring to it, to determine where each metal goes at every stage of the analysis. *This thought should be kept constantly in mind in making the record.* It is not necessary, however, to repeat statements from the text-book and the more concise the record can be made the better, provided it contains all essential facts and is clear. An illustration will, perhaps, be of service.

Silver.

$$AgNO_3+HCl=\underset{\text{White pr.}}{\underline{AgCl}}+HNO_3.$$

$AgCl$+hot water—does not dissolve, since the filtrate gives no pr. with H_2S.

$$2\,AgCl+3NH_4OH=\underset{\text{Clear sol.}}{\underline{2\,AgCl.3\,NH_3}}+3H_2O.$$

In recording an analysis, the following form has been found convenient by the author:

No.	Substance.	Reagent.	Result.	Inference.	Precip.	Solution.
1	Colorless Sol.	HCl.	Wh. pr.	Gr. I.	$AgCl.\ HgCl.PbCl_2$.	
2	Pr. 1.	Hot H_2O.	Partial sol.		$AgCl.\ HgCl$.	$PbCl_2$.
3	Sol. 2.	H_2SO_4.	Wh. pr.	Pb.	$PbSO_4$.	
4	Pr. 2.	NH_4OH.	Blackens.		Hg_2NH_2Cl.	$2\ AgCl.\ 3NH_3$.
5	Sol. 4,	HNO_3.	No pr.	No Ag.		

The numbers in the second column refer to the operation which gave the precipitate or solution referred to, and any metal can be followed by these numbers from the beginning to the end of the analysis. The columns headed "Precip." and "Solution" give the compounds which would be present in each case if all metals of the group were present.

The student will find it convenient to number his record in accordance with the statements given below for each metal. *It is important that the record should be made in the laboratory as the experiments are performed.*

GROUP I.

Lead.

1. Lead nitrate and hydrochloric acid give?
2. Lead chloride and hot water.
3. Lead chloride and hydrogen sulphide give?
4. Lead chloride and sulphuric acid give?
5. Lead chloride. potassium pyrochromate and ?, give lead chromate, chromic acid and?

Silver.

1. Silver nitrate and hydrochloric acid give?
2. Silver chloride and hot water.
3. Silver chloride and ammonia give?
4. Ammonio-silver chloride and nitric acid give?
5. Silver chloride exposed to the light.

Murcury in Mercurous Salts.

1. Mercurous nitrate and hydrochloric acid give?
2. Mercurous chloride and hot water.
3. Mercurous chloride and ammonia give ammonium chloride and?
4. Mercurous-amido-chloride and aqua regia give mercuric chloride, ammonium chloride, nitric oxide and water. Write first a reaction between mercurous-amido-chloride and hydrochloric acid giving mercuric chloride, ammonium chloride and hydrogen; then a reaction between hydrogen

and nitric acid giving nitric oxide and water; then combine the two equations so as to eliminate free hydrogen.

5. Mercuric chloride and stannous chloride give stannic chloride and?

GROUP II.

Sub-Group A.

Mercury in Mercuric Salts.

1. Mercuric chloride and hydrogen sulphide give?
2. Mercuric sulphide and nitric acid.
3. Mercuric Sulphide and aqua regia give mercuric chloride, nitric oxide, sulphur and water. Write on the same principle as mercurous-amido chloride and aqua regia.
4. Mercuric chloride and stannous chloride give?

Lead.

1. Lead chloride and hydrogen sulphide give?
2. Lead sulphide and nitric acid give lead nitrate, nitric oxide, sulphur and water. Write first lead sulphide and nitric acid give lead nitrate and hydrogen sulphide; then hydrogen sulphide and nitric acid give nitric oxide, sulphur and water; then combine the two reactions.
3. Lead nitrate and sulphuric acid give ?
4. Lead sulphate and ammonium tartrate form, probably, a soluble, double salt. Write the statement without a reaction.
5. Lead sulphate and potassium pyrochromate give?

Bismuth.

1. Bismuth chloride and water give ?
2. Bismuth oxychloride and hydrogen sulphide give?
3. Bismuth sulphide and nitric acid give?
4. Bismuth nitrate and sulphuric acid give? Be careful to record whether there is a precipitate or not.
5. Bismuth sulphate and ammonia give?

6. Bismuth hydroxide and hydrochloric acid give?
7. Bismuth chloride and water give?

Copper.

1. Copper sulphate and hydrogen sulphide give?
2. Copper sulphide and nitric acid give?
3. Copper nitrate and sulphuric acid give?
4. Copper sulphate and ammonia give?
5. Cupric ammonium sulphate and hydrochloric acid give copper sulphate and?
6. Copper sulphate and iron give?

Cadmium.

1. Cadmium nitrate and hydrogen sulphide give?
2. Cadmium sulphide and nitric acid give?
3. Cadmium nitrate and sulphuric acid give?
4. Cadmium sulphate and ammonia give?
5. Cadmium hydroxide and hydrochloric acid give?
6. Cadmium chloride and iron.
7. Cadmium chloride and hydrogen sulphide.

Sub-Group B.

Arsenic in Arsenious Compounds.

1. Arsenious oxide and hydrogen sulphide give?
2. Arsenious sulphide and ammonium sulphide give?
3. Ammonium sulpharsenite and hydrochloric acid give ammonium chloride and?
4. Arsenious sulphide and con. hydrochloric acid.
5. Arsenious sulphide, potassium chlorate, hydrochloric acid and water give arsenic acid, potassium chloride, hydrochloric acid and sulphur. Write:
    - *a.* Potassium chlorate and hydrochloric acid give?
    - *b.* Chloric acid gives hydrochloric acid and oxygen.
    - *c.* Arsenious sulphide, oxygen and water give arsenic acid and sulphur.

Combine *a* and *b* to eliminate chloric acid, and the resulting equation with *c*.

6. Arsenic acid, magnesium sulphate and ammonia give?

Arsenic in Arsenic Compounds.

1. Arsenic acid and hydrogen sulphide give?
2. Arsenious oxide, potassium pyrochromate, hydrochloric acid and water give arsenic acid, chromic chloride and potassium chloride.
3. Arsenic acid, magnesium sulphate and ammonia give?

Tin.

1. Stannous chloride and hydrogen sulphide give?
2. Stannous sulphide and ammonium polysulphide, $(NH_4)_2S_2$, give stannic sulphide and ammonium sulphide. The stannic sulphide then dissolves as a sulpho-salt.
3. Ammonium sulphide, stannic sulphide and hydrochloric acid give?
4. Stannic sulphide and con. hydrochloric acid give?
5. Stannic chloride and iron give?
6. Stannous chloride and mercuric chloride give?

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1. Stannic chloride and hydrogen sulphide give?
2. Stannic chloride and mercuric chloride.

Antimony.

1. Antimony chloride and hydrogen sulphide give?
2. Antimony sulphide and ammonium sulphide give? The antimony sulphide dissolves as a sulpho-salt. Write the statement without a reaction.
3. Antimony sulphide, ammonium sulphide and hydrochloric acid give?
4. Antimony sulphide and con. hydrochloric acid give?
5. Antimony chloride and iron give?
6. Antimony chloride and zinc give?

Experiments with gold and platinum are usually omitted.

GROUP III.

Chromium.

1. Chrome alum and ammonia give?
2. Chromic hydroxide and hydrochloric acid give?

3. Chromic chloride and barium carbonate give a basic salt of chromium and? The filtrate should contain no chromium, but may give a slight precipitate of barium carbonate.

4. The basic salt of chromium with caustic soda gives chromic hydroxide; does the latter dissolve or not?

5. Chromic hydroxide, sodium carbonate and potassium chlorate give potassium chromate, salt, carbon dioxide, water and oxygen.

Potassium pyrochromate, hydrochloric acid and hydrogen sulphide give sulphur and?

Aluminium.

1. Alum and ammonia give?
2. Aluminium hydroxide and ammonium sulphide.
3. Aluminium hydroxide and hydrochloric acid give?
4. Aluminium chloride and barium carbonate give aluminium hydroxide and?
5. Aluminium hydroxide and caustic soda give?
6. Sodium aluminate and hydrochloric acid give?
7. Aluminium chloride and ammonia give?

Iron.

1. Ferric chloride and ammonia give?
2. Ferric hydroxide and ammonium sulphide give?
3. Ferrous sulphide and hydrochloric acid give?
4. Ferrous chloride, nitric acid and hydrochloric acid give ferric chloride, nitric oxide and water.
5. Ferric chloride and potassium sulpho-cyanate give?
6. Ferric chloride and potassium ferrocyanide give?
7. Ferric chloride and barium carbonate give a basic salt and?

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1. Ferrous sulphate and potassium sulpho-cyanate should give no color.
2. Ferric sulphate and potassium sulpho-cyanate give?
3. Ferrous sulphate and potassium ferrocyanide give?

4. Ferric sulphate and potassium ferrocyanide give?
5. Ferrous sulphate and potassium ferricyanide give?
6. Ferric sulphate and potassium ferricyanide give?
7. Ferrous sulphate and caustic soda give?
8. Ferric sulphate and caustic soda give?
9. Ferrous sulphate, sulphuric acid and nitric acid give?
10. Ferric sulphate and hydrogen sulphide give?

Cobalt.

1. Cobalt nitrate, ammonium chloride and ammonia. Try also with very little ammonia and no ammonium chloride.
2. Cobalt nitrate and ammonium sulphide give?
3. Cobalt sulphide and dil. hydrochloric acid.
4. Cobalt sulphide and aqua regia give? See mercury.
5. Cobalt chloride and caustic soda give?
6. Cobalt hydroxide and acetic acid give? Acetic acid is mono-basic.
7. Cobalt acetate, potassium nitrite, and acetic acid give potassium cobaltic nitrite, potassium acetate, nitric oxide (2 molecules) and water.
8. Cobalt compounds in the borax bead.

Nickel.

1–6. As with cobalt.

7. Nickel acetate and potassium nitrite.
8. Nickel acetate and caustic soda give?
9. Nickel compounds in the borax bead.

Manganese.

1. Manganese sulphate, ammonium chloride and ammonia. Try also without ammonium chloride.
2. Manganese sulphate and ammonium sulphide give?
3. Manganese sulphide and hydrochloric acid give?
4. Manganese chloride and barium carbonate.
5. Manganese chloride and ammonium sulphide give?
6. Manganese sulphide and hydrochloric acid give?
7. Manganese chloride and caustic soda give?
8. Manganese hydroxide, sodium carbonate and oxygen give sodium manganate, carbon dioxide and water.

Zinc.

1—6. As with manganese.

7. Zinc chloride and caustic soda give sodium zincate, ZnO_2Na_2, and?

8. Sodium zincate and hydrogen sulphide give?

GROUP IV.

Barium.

1. Barium chloride and ammonium carbonate give?
2. Barium carbonate and acetic acid give?
3. Barium acetate and calcium sulphate give?
4. Barium acetate and potassium pyrochromate give?
5. Barium chromate and hydrochloric acid give?
6. Barium chloride and sulphuric acid give?

Strontium.

1—3. As with barium.

4. Strontium acetate and potassium pyrochromate.
5. Strontium acetate and ammonium carbonate give?
6. Strontium carbonate and acetic acid give?
7. Strontium acetate and calcium sulphate give?
8. Strontium acetate and ammonium sulphate give?

Calcium.

1—8. As with strontium.

9. Calcium sulphate and ammonium oxalate give?

GROUP V.

Magnesium.

1. Magnesium chloride, ammonia and sodium phosphate give?
2. Magnesium chloride and barium hydroxide give?

Sodium.

1. Flame color of sodium.
2. Flame color of sodium and potassium.

Potassium.

1. Flame color of potassium.
2. Chloro-platinic acid and potassium chloride give?

Ammonium.

1. Chloro-platinic acid and ammonium chloride give?
2. Effect of heat on ammonium salts.
3. Ammonium chloride and caustic soda give?

REAGENTS.

Dil. HCl. 1 : 4 by vol. Contains about 8.5 per cent. HCl.

Dil. HNO_3. 1 : 3 by vol. Contains about 20 per cent. HNO_3.

HNO_3. For alloys 1 : 1 by vol. Contains about 40 per cent. HNO_3.

Dil. H_2SO_4. 1 : 5 by vol. Contains about 25 per cent. H_2SO_4.

Acetic acid. Sp. gr. 1.04. Contains about 30 per cent. $C_2H_4O_2$.

H_2S. Best from FeS and com'l HCl, diluted 1 : 1.

Chlorine water. Keep in a bottle painted black.

$NaOH$. 1 : 10. Allow to settle and siphon off.

NH_4OH. Sp. gr. 96. Contains about 10 per cent. NH_3.

BaO_2H_2. 1 : 20. Use hot water.

CaO_2H_2. Digest slaked lime with water and filter.

$(NH_4)_2S$. Pass the gas evolved from the action of 360 grams (200 cc.) of con. com'l H_2SO_4 properly diluted, upon an excess of FeS (calculated about 320 grams), into one litre of NH_4OH. If yellow $(NH_4)_2S$ is desired, add a little flowers of sulphur.

Na_2HPO_4. 1 : 10. Use cryst. salt and warm water.

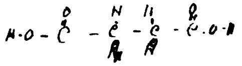
Na_2CO_3. 1 : 2. Use cryst. salt and warm water, or 1 : 5, using the dry salt.

$(NH_4)_2CO_3$. 200 grams "Am. Carbonate."
200 grams (209 cc.) NH_4OH.
800 cc. water.
} Digest in a closed bottle.

$(NH_4)_2C_2O_4$. 1 : 25. Use warm water.

$(NH_4)_2SO_4$. 1 : 10.

NH_4Cl. 1 : 10.



$(NH_4)_2C_4H_4O_6$ Dissolve 100 grams of tartaric acid in 250 cc. of NH_4OH and dilute to one litre. The solution should be slightly alkaline.

$NH_4C_2H_3O_2$. Add 100 cc. of acetic acid to 96 cc. of NH_4OH and exactly neutralize with NH_4OH or $C_2H_4O_2$.

$(NH_4)_2MoO_4$. Dissolve 100 grams of MoO_3 in 400 grams (417 cc.) of NH_4OH, filter and pour slowly with constant stirring into 1500 grams (1250 cc.) of HNO_3 of sp. gr. 1.20. Keep for several days in a warm place and decant or filter.

$K_2Cr_2O_7$. 1 : 10. Use warm water.
K_4FeCy_6. 1 : 15.
KCNS. 1 : 15.
KCN. 1 : 5. Use cold water. Does not keep well.
K_3FeCy_6. The solution does not keep. Use the powder.
KNO_2. 1 : 4. Neutralize with $C_2H_4O_2$.
$BaCl_2$. 1 : 10.
$BaCO_3$. Mix with about 10 parts of water.
$CaCl_2$. 1 : 5. Use the cryst. salt.
$CaSO_4$. Saturated solution.
$MgSO_4$. 1 : 10.
Fe_2Cl_6. 1 : 10. Use the cryst. salt.
$Co(NO_3)_2$. 1 : 15. Use the cryst. salt.

$FeSO_4$. 12 grams cryst. $FeSO_4$.
6 grams $(NH_4)_2SO_4$.
2 cc. con. H_2SO_4.
100 cc. water.
} The solution will keep well enough to use in tests for HNO_3 for several months.

$AgNO_3$ 1 : 40.
$Pb(C_2H_3O_2)_2$. 1 : 20.
$PtCl_4$. 1 : 50.
$SnCl_2$. Dissolve tin in conc. HCl, dilute with four volumes of water slightly acidified with HCl, and keep in a bottle containing granulated tin.
Alcohol.
Carbon disulphide.

DRY REAGENTS.

$KClO_3$.
$K_3FeC_6N_6$.
$K_2CO_3+Na_2CO$. 4:3.
$KHSO_4$.
KCN.
Na_2CO_3.
$Na_2B_4O_7 + 10\ H_2O$
$NaNH_4HPO_4 + 8\ H_2O$.
NH_4Cl.
$CaCO_3$.
MnO_2.
Iron nails.
Zinc.
Platinum.
Litmus paper.
Turmeric paper.
Beeswax.
Starch.

TABLE OF COMPOUNDS.

The table below gives the compounds of each metal which are formed in the course of an analysis. It may be followed as a guide in analysis by any one who is familiar with the details involved. It may also be of service to the beginner in gaining a clear conception of the analytical processes.

Gr. I.	$PbCl_2$	$PbCrO_4$								
	$HgCl$	Hg_2NH_2Cl		$HgCl_2$	$HgCl$					
	$AgCl$	$2AgCl3NH_3$		$AgCl$						
Gr. II.	HgS	HgS	$HgCl_2$	$HgCl$						
	PbS	$Pb(NO_3)_2$		$PbSO_4$	—	$PbCrO_4$				
	Bi_2S_3	$Bi(NO_3)_3$	$Bi_2(SO_4)_3$		$BiOOH$	$BiCl_3$	$BiOCl$			
	CuS	$Cu(NO_3)_2$	$CuSO_4$		$CuSO_44NH_3$		$CuCl_2$	Cu		
	CdS	$Cd(NO_3)_2$	$CdSO_4$		$Cd(OH)_2$		$CdCl_2$	$CdCl_2$	CdS	
	As_2S_3	As_2S_3	H_3AsO_4		$MgNH_4AsO_4$					
	Sb_2S_3	$SbCl_3$		Sb	Sb					
	SnS_3	$SnCl_4$		Sn	$SnCl_2$	$SnCl_4[HgCl]$				
Gr. III.	CoX_2	CoS	CoS	$CoCl_2$	$Co_2(NO_2)_6.(KNO_2)_6$. Blue bead.					
	NiX_2	NiS	NiS	$NiCl_2$	$Ni(C_2H_3O_2)_2$	$Ni(OH)_2$. Reddish brown bead.				
	$Fe_2(OH)_6$	FeS	$FeCl_2$	$FeCl_3$	Basic salt	$Fe_2(OH)_6$	Fe_2O_3			
	$Cr_2(OH)_6$	$Cr_2(OH)_6$	$CrCl_3$	$CrCl_3$	Basic salt	$Cr_2(OH)_6$	K_2CrO_4			
	$Al_2(OH)_6$	$Al_2(OH)_6$	$AlCl_3$	$AlCl_3$	$Al_2(OH)_6$	Na_3AlO_3	$AlCl_3$	$Al_2(OH)_6$		
	MnX_2	MnS	$MnCl_2$	$MnCl_2$	$MnCl_2$	MnS	$MnCl_2$	$Mn(OH)_2$	Na_2MnO_4	
	ZnX_2	ZnS	$ZnCl_2$	$ZnCl_2$	$ZnCl_2$	ZnS	$ZnCl_2$	$ZnCl_2$	ZnO_2Na_2	ZnS
Gr. IV.	$BaCO_3$	$Ba(C_2H_3O_2)_2$		$BaSO_4$		$BaCrO_4$	$BaCl_2$	$BaSO_4$		
	$SrCO_3$	$Sr(C_2H_3O_2)_2$		$SrSO_4$		SrA_2	$SrCO_3$	SrA_2	$SrSO_4$	
	$CaCO_3$	$Ca(C_2H_3O_2)_2$		$Ca(C_2H_3O_2)_2$		CaA_2	$CaCO_3$	CaA_2	CaA_2	CaC_2O_4
Gr. V.	$MgNH_4PO_4$		MgX_2		$Mg(OH)_2$					
					BaX_2	$BaCO_3$				
	NH_4X		Volatilized. —			NH_4X	Volatilized.			
	KX		KX		KX	KX	KX	K_2PtCl_6		
	NaX		NaX		NaX	NaX	NaX	NaX—Yellow flame.		

Table of

	Arsenate.	Arsenite.	Chromate.	Sulphate.	Phosphate.	Borate.	Oxalate.	Fluoride.	Carbonate.	Silicate.
Silver	HNO_3	HNO_3	HNO_3	W-A	HNO_3	HNO_3	HNO_3	W	HNO_3	
Mercury (ous)	HNO_3	HNO_3	HNO_3	W-A	HNO_3		HNO_3		HNO_3	
Mercury (ic) .	HCl	HCl	W-A	W-A	HCl		HCl	W-A	HCl	
Lead	HNO_3	HNO_3	A-I	A-I	HNO_3	HNO_3	HNO_3	HNO_3	HNO_3	HNO_3
Bismuth . . .	HCl		HCl	W	HCl	HCl	HCl	W	HCl	
Copper	HCl	HCl	W	W	HCl	HCl	HCl	HCl	HCl	HCl
Cadmium . . .	HCl		HCl	W	HCl	W-A	HCl	W-A	HCl	HCl
Tin (ous) . . .	HCl	HCl	HCl	W	HCl	HCl	HCl	W		
Tin (ic)	HCl				HCl		W	W		
Antimony . .	HCl	HCl	HCl	HCl	W-A		HCl	W		
Iron (ous) . .	HCl	HCl		W	HCl	HCl	HCl	W-A	HCl	HCl
Iron (ic) . . .	HCl	HCl	W	W	HCl	HCl	HCl	W	HCl	HCl
Aluminium . .	HCl			W	HCl	HCl	HCl	W		A-I
Chromium . .	HCl		HCl	W	HCl	HCl	W-A	W		HCl
Cobalt	HCl	HCl	HCl	W	HCl	HCl	HCl	W-A	HCl	HCl
Nickel	HCl	HCl	HCl	W	HCl	HCl	HCl	W-A	HCl	HCl
Manganese .	HCl	HCl	W	W	HCl	HCl	W-A	HCl	HCl	HCl
Zinc			W	W	HCl	HCl	HCl	W-A	HCl	HCl
Barium . . .	HCl	HCl	HCl	I	W & A	HCl	HCl	A-I	HCl	HCl
Strontium . .	HCl	HCl	W-A	I	HCl	HCl	HCl	A-I	HCl	HCl
Calcium . . .	HCl	HCl	W-A	W-I	W & A	HCl	HCl	A-I	HCl	HCl
Magnesium . .	HCl	HCl	W	W	HCl	W-A	HCl	A-I	HCl	HCl
Potassium . .	W	W	W	W	W	W	W	W	W	W
Sodium . . .	W	W	W	W	W	W	W	W	W	W
Ammonium .	W	W	W	W	W	W	W	W	W	

HNO_3. Soluble in HNO_3, in most cases insoluble, or slightly soluble in HCl.
HCl. Soluble in HCl, in many cases soluble in other acids, also.
W. Soluble in water.

Solubilities.

Chloride.	Bromide.	Iodide.	Cyanide.	Ferrocyanide.	Ferricyanide.	Sulphide.	Nitrate.	Chlorate.	Acetate.	Tartrate.	Citrate.	Oxide.	Hydroxide.
I	I	I	1	I	I	HNO_3	W	W	W	HNO_3	HNO_3	HNO_3	
A-I	A-I	HNO_3				Aq.Reg	W	W	W-A	W-A	HNO_3	HNO_3	
W	W	HNO_3	W			Aq.Reg	W	W	W	HCl	W-A	HCl	
W-I	W-I	W-A	HNO_3	A	W-A	HNO_3	W	W	W	HNO_3	HNO_3	HNO_3	HNO_3
W-A	W-A	HCl				HNO_3	W-A	W	W	HCl		HCl	HCl
W	W	W	HCl	I		HNO_3	W	W	W	W	W	HCl	HCl
W	W	W	HCl			HNO_3	W	W	W	W-A	HCl	HCl	HCl
W		W				HCl		W	W	HCl		HCl	HCl
W		W				HCl			W			A&I	HCl
W-A	W-A	W-A				HCl				A&W		HCl	HCl
W	W	W	A-I	I	I	HCl	W	W	W	W-A	W	HCl	HCl
W	W	W		1	W	Aq.Reg	W	W	W	W	W	HCl	HCl
W	W	W				HCl	W	W	W	W	W	A&I	HCl
W & I	W & I	W	HCl			A-I	W	W	W	W	W	A&I	HCl
W	W	W	A-I	I	I	HNO_3	W	W	W	W	W	HCl	HCl
W	W	W	A-I	I	I	HNO_3	W	W	W	HCl	W	HCl	HCl
W	W	W	HCl	HCl	I	HCl	W	W	W	W-A	HCl	HCl	HCl
W	W	W	HCl	A-I	HCl	HNO^3	W	W	W	HCl	W-A	HCl	HCl
W	W	W	W-A	W-A		W	W	W	W	HCl	HCl	W	W
W	W	W	W	W		W	W	W	W	HCl	HCl	W	W
W	W	W	W	W	W	W-A	W	W	W	HCl	W-A	W&A	W-A
W	W	W	W	W	W	HCl	W	W	W	W-A	W	HCl	HCl
W	W	W	W	W	W	W	W	W	W	W	W	W	W
W	W	W	W	W	W	W	W	W	W	W	W	W	W
W	W	W	W	W	W	W	W	W	W	W	W		W

W-A. Slightly soluble in water, soluble in acids.
A-I. Insoluble in water, slightly soluble in acids.
I. Insoluble in water and acids.

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